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DEFORMATION AND ANNEALING STUDY OF NICRALY

by

D. M. TRELA and L. J. EBERT

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School of Engineering

Department of Metallurgy and Materials Science

Case Western Reserve University

Cleveland, Ohio 44106



ABSTRACT

Extensive experiments were carried out on the ODS alloy "Nicaly", (an alloy prepared by mechanical alloying and consolidating a powder blend consisting of 16% chromium, 4% aluminum, 2-3% yttria, balance nickel), in efforts to develop methods of controlling the grain size and grain shape of the material. The experiments fell into two general categories: variations in the annealing parameters using the as-extruded material as it was received, and various thermo-mechanical processing schedules (various combinations of cold work and annealing). The latter experiments were patterned after those which were successful in developing controls for grain size and grain shape in both TD-Nickel and TD-Nichrome.

Success was achieved in gaining grain size and grain shape control by the first of the control systems (annealing of the as-extruded material). By proper selection of annealing temperature and cooling rates, the grain size of the as-received material could be increased almost two orders of magnitude (from an average grain dimension of 0.023 mm to 1.668 mm) while the aspect ratio could be increased by some 50% (from 20:1 to 30:1).

While the experiments are not yet quite complete, no success has been achieved to date in gaining significant control of the grain size and shape of the material by thermo-mechanical processing. Longitudinal cold rolling of the fully annealed material can be used to

increase the aspect ratio of the grains by some 50%. However, the cold-worked material does not have a stable grain size at temperatures beyond 1600°F (871°C) because recrystallization begins to occur at temperatures slightly higher than this.

The very sluggish nature of the recrystallization kinetics, while giving some stability to the cold worked material, appears to preclude the use of the recrystallization mechanism to achieve very large grain sizes, the system which was used to very great success for the predecessor ODS materials, TD-Nickel and TD-Nichrome. When the cold worked Nicraly does recrystallize, it does so with an exceedingly fine grain, equiaxed in shape. The recrystallized grains are almost immune to significant grain growth, even at temperatures within 100°F (56°C) of the melting temperature range of the alloy. Further, it is very difficult, if not impossible, to achieve 100% recrystallization of the cold worked material, even when the material is cold worked severely (73% reduction in thickness by rolling) and annealed at very high temperatures (2450°F, 1343°C).

Work planned for the coming year includes completion of the thermo-mechanical processing experiments (utilization of combinations of longitudinal and cross rolling prior to annealing, and the use of critical reductions to attempt to produce abnormal grain growth), generation of the high temperature properties (tensile, creep, and rupture) of the material with several different grain sizes and shapes, measurement of the thermodynamic characterization of the high temperature deformation process for the material, and the testing of the

previously developed theoretical model for the high temperature deformation of ODS alloys with the new Nicrally material. This last item is a continuation of the efforts to develop and validate a completely general theoretical model for high temperature deformation of all ODS alloys, which should find use in the design of future alloys of this generic type.

FOREWORD

This Annual Status Report represents investigations conducted under NASA Grant NGR 36-003-094 from July 1974 to June 1975. The initial portion of this presentation describes the work presented to Case Western Reserve University by David M. Trela in partial fulfillment of the requirements for the degree of Master of Science.

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DEFORMATION AND ANNEALING STUDY OF NICRALY

INTRODUCTION

The proven validity of the concept of oxide-dispersion strengthening which the development of SAP (Sintered Aluminum Powder) provided almost a quarter century ago has led to a continuing, although sporadic, research and development activity aimed at the production of the "ultimate" high temperature alloy. The "target" alloy is one which embodies maximum strength and creep resistance at temperatures well above one-half T_m , metallurgical stability at high use temperatures, oxidation and corrosion resistance in high velocity streams of hot gasses (including air), adequate formability at room temperature, acceptable machinability, thermal shock resistance, and minimum density, consistent with the other requirements.

In the absence of a complete understanding of the basic principles which govern the high temperature properties of the oxide-dispersion strengthened alloys, empiricism was used in the development of each of the "generations" of alloys. Once the alloys were available, the basic research efforts expended upon them were generally successful in rationalizing the basic principles underlying the experimentally determined behaviors. Thus, with the creation of each new generation of alloys, and the subsequent basic research study of the performance of that generation, the general theory of dispersion-oxide strengthening has begun to take shape. The ultimate goal of this evolution is the development of a complete and sufficient under-

standing of the role of various factors which control the high temperature behavior and characteristics of oxide-dispersion strengthened materials, so that future development of the materials may be based upon theory, rather than empiricism. In addition to being more efficient than empiricism, theory based development will permit the "tailoring" of materials to fit specific requirements.

Besides providing engineeringly important evaluation data on high temperature performance of the various generations of oxide-dispersion strengthened alloys, it is the objective of the study research project, the latest findings of which are reported upon herein, to make significant contributions to the evolution of the complete theory of oxide dispersion strengthening. The present efforts of the study are being directed at the third of the modern generations of the alloys, the nickel-base alloy containing nominally 16 percent chromium, 4 percent aluminum, with 2-3 percent of yttria (Y_2O_3) as the disperse oxide addition. The current research activities utilize all of the findings of the studies of the previous alloys, including those generated by other investigators in the field.

As noted above, the basic research study of each of the generations of oxide-dispersion strengthened alloys (ODS alloys) contributed to the fund of basic principles upon which the theory of oxide dispersion strengthening is being built. Although early formulations of alloys had to be abandoned when various shortcomings became apparent, the basic principles were applied to the succeeding formulations.

Work on the first of the modern ODS alloys, TD-Nickel,⁽¹⁻⁸⁾, demonstrated that grain size (volume), grain shape, and grain orientation played major roles in the creep resistance of the alloy. Also, it provided the basis of the present "hole theory" for the role of the dispersoid oxide phase in the strengthening mechanism. Further, it revealed methods by which grain size and shape could be controlled, and for the first time provided a rationale for the previously unexplained recrystallization mechanics of TD-Nickel. Thus, despite the abandonment of the alloys because of limitation of low intrinsic strength and poor oxidation resistance, these theoretical findings were applied to the next in the series of alloys, TD-Nickel-Chromium.

The TD-Nickel-Chromium alloys were designed to overcome the shortcomings of the TD-Nickel alloys, in that the chromium added to improve both the intrinsically low strength of the pure nickel matrix, and the poor oxidation resistance of nickel. From a basis understanding point of view, they constituted a vehicle with which to assess the findings from the study on TD-Nickel.

Work on the TD-Nickel-Chromium⁽⁹⁾ verified the importance of grain size and shape in achieving good creep resistance. While the material was not as responsive to grain size control techniques developed for TD-Nickel as was the TD-Nickel, it was sufficiently amenable to thermo-mechanical processing prior to final anneal as to give favorable grain conformations. More importantly, however, the grain size and shape control techniques provided a material with which to develop a model for the creep-deformation mechanism of ODS

alloys, particularly since some of the material was available as single crystals. The "concurrent" model for the creep deformation, involving both grain deformation and grain boundary sliding, with a different temperature dependence for each mode, seems to be a completely rational and viable model with which to explain the creep deformation of all ODS alloys with single-phase matrices. Further, it is expected that it will be at least adequate, if not completely sufficient, to rationalize or predict the creep resistance propensity of ODS alloys with two or more thermodynamically compatible phases in the matrix. Hence, despite the loss of favor of the TD-Nickel-Chromium alloys because of their poor oxidation resistance in the presence of high velocity flowing gasses, they too have served an important purpose in the present study.

The current generation of ODS alloys, those containing nominally 16 percent chromium, 4 percent aluminum, and 2-3 percent yttria, in a nickel base, are the subject of the present investigation on the overall project. They have been shown to be capable of overcoming the deficiencies of the predecessor TD-Nickel-Chromium, in that they have adequate oxidation resistance to high velocity flowing gasses, while (apparently) retaining the high temperature creep resistance of TD-Nickel-Chromium.

This report summarizes the work performed on the ODS alloy, Ni-16Cr-4Al-Y₂O₃ (Nicaloy), during the grant award period 1 July 1974 to 30 June 1975. Unfortunately, because of delays in receiving material with which to conduct the study, the extent of the progress is some-

what less than had been anticipated for the period.*

*Material for the study was not received until late in 1974. Pending receipt of the material, work on the project was concerned with the finalization of the results of the work on TD-Nickel-Chromium.

PLAN FOR THE INVESTIGATION

The basic philosophy of the approach to the study of the Nicraly was one of applying the results of the studies on TD-Nickel and TD-Nickel-Chromium to Nicraly in an effort to achieve the most favorable grain size and shape for creep resistance at high temperatures and high stresses, and then measuring the creep characteristics of the material with the optimum grain configuration and shape. Simultaneously, efforts were to be directed at testing the theoretical model for the creep deformation mechanism on this new material, and modifying it as necessary. The ultimate goal of this part of the effort was the development, and validation, of a comprehensive (or universal) model for the behavior of ODS materials.

From a practical standpoint, the Nicraly appeared to be the best of the ODS alloys developed to date. Even with no particular efforts to control grain size and shape, the material was found to have very high strength and creep resistance at 2000°F and higher. As a matter of fact, it was found to have usable strengths to within 100°F of its melting point. Further, it was found to have (at least) adequate oxidation resistance under intended use conditions.

From a fundamental ODS materials standpoint, Nicraly is an interesting material with which to continue the development of a "universal" model for the creep resistance behavior of ODS materials. Its highly alloyed matrix, which can be either single-phase or two-phase (depending upon the heat treatment given to the material), should

permit the increase in the amount of stored strain energy which can be added to the alloy. This stored energy is the driving force for recrystallization in most alloys, and it was this recrystallization process which gave the grain size control in TD-Nickel and TD-Nickel-Chromium. On the other hand, the highly alloyed nature of the matrix would impede the diffusion processes, and thus might act in a way as to reduce recrystallization rates.

Further from the standpoint of basic fundamentals, the highly alloyed (stiff) nature of the matrix of Nicraly offers a potential solution to the problem of grain deformation (by dislocation motion mechanisms) at high creep temperatures. It was this grain deformation which was found to be one of the limiting factors in the creep resistance of TD-Nickel-Chromium at temperatures in excess of 2000°F. In other words, despite the absence of, or the minimization of the amount of, grain boundary material in TD-Nickel-Chromium (and the concomitant minimization of grain boundary sliding), the presence of the thorium particles within the grains of the solid solution matrix of nichrome did not impart sufficient resistance to dislocation motion within the grains to give good creep resistance in the TD-Nickel-Chromium at temperatures in the order of 2200°F and higher. It was postulated that the combined strengthening effects of the oxide disperse phase and the higher strength matrix might achieve the desired resistance to dislocation motion. This would mean that the material should have superior creep resistance at 2200-2400°F, if the grain size were sufficiently large.

In brief, the plan of attack used in this study might be summarized as the following:

1. Assess the effectiveness of proven methods of attaining large grain size and aspect ratio in TD-Nickel and TD-Nickel-Chromium alloys as they relate to the Nicrally.
2. Produce Nicrally with optimum grain size and shape, using the techniques developed in (1) above.
3. Measure high temperature tensile and creep properties of material with optimum grain size and shape, and with grain sizes and shapes somewhat different than those considered to be optimum.
4. Measure activation enthalpies for the material in tension and in creep as a function of grain size, temperature and stress level, to assess the contributions of the several deformation mechanisms which are operative in the material.
5. Evaluate the relative roles of deformation modes in terms of the theoretical model developed in earlier work, in an attempt to test the model for its "universality".
6. Adjust the model as necessary on the basis of the results of (5) above, generating additional data as indicated.

As noted above, the time delay in procuring the necessary material with which to start the investigation has limited the results to item (1) above.

MATERIALS AND PROCEDURES

Material

As noted above, the Nicraly material being studied is a nickel-base alloy, with nominally 16% chromium, 4% aluminum, and 2-3% Y_2O_3 in the powders which are mechanically attritted, and consolidated by hot extrusion. The material was supplied by the Stellite Division of Cabot Corporation, as arranged by personnel of NASA Lewis, through Dr. R. Grierson of Cabot.

Because of certain uncontrollable factors, delays were encountered in the receipt of the materials for the study. Further, two different grades of the nominal composition material were supplied. However, the preferred material did not become available until some time after that at which the second material was in-hand. As a result, in order to expedite progress on the study, it was necessary to begin the study with a grade of the composition which would not ordinarily have been used. Thus, the bulk of the work was carried out on this material.

The chemical compositions for the two different grades of the material are shown below:

<u>ELEMENT</u>	<u>QUANTITY - Wt. %</u>	
	<u>Powder Batch 101*</u>	<u>Powder Batch 204B**</u>
Aluminum	3.08	3.95
Carbon	0.091	0.05
Chromium	15.19	15.8
Iron	ND***	1.31
Nickel	77.91	76.20
Yttrium	0.58	0.99
Oxygen	ND***	4810 ppm

* Semi-quantitative spectrographic analysis of sample supplied by investigator to Crobaugh Laboratories, Cleveland, Ohio

** Analysis supplied by Dr. Grierson (Cabot) to investigator.

*** Not determined.

Because of the difference in the type of the analytical procedures used for the two different grades of materials, no significance can be attributed to the difference in the yttrium content of the two batches. The material made from powder batch 204B is the preferred material; however, as noted above, it was necessary to perform the bulk of the work to date on the material from powder batch 101.

The material was supplied in the "as-hot extruded" condition, with the extrusion being carried out at 1900°F with a 14.14:1 extrusion

ratio. Material produced from both powder batches was supplied in the form of the two bars, the dimensions of which are listed below:

Powder Batch 101: 2 pieces, each $3/4"$ x $2\frac{1}{2}"$ x $12"$ long

Powder Batch 204B: 1 piece, $3/4"$ x $2\frac{1}{2}"$ x $24"$ long

1 piece, $3/4"$ x $2\frac{1}{2}"$ x $7\frac{1}{2}"$ long

For each bar, the long dimension was reported to being parallel to the extrusion direction. Figure 8 shows the basic directions being described here.

Rolling Deformation Study

Deformation prior to the annealing study was carried out by cold rolling on a Fenn 2-Hi 75 h.p. mill, equipped with 10 inch diameter rolls. All samples to be rolled were nominally 1 inch long in the rolling direction, $3/4$ inch wide, and less than $3/8$ inch thick. Thicker samples could not be accommodated without damaging the rolls. The use of the thinner samples assured an equal amount of deformation throughout the sample.

Preparatory to rolling, the sawed blanks were first annealed by heating them at 1343°C (2450°F) for one hour in argon and then air cooling. The intent of this pre-rolling treatment was several-fold. It provided additional chemical homogenization; it softened the material to allow maximum cold working; it yielded a large grain (starting) grain size. This third factor was an incidental benefit, which was found during the annealing study (described later).

The 101 material allowed 73 percent reduction by longitudinal rolling before cracking occurred. Up to 15 percent reduction by cross rolling was possible before cracking in the 204B material. Large reductions per pass, and correspondingly, few passes were necessary to avoid splitting and cracking. The rolling mill could accommodate only 10 percent reductions per pass, and still avoid possible damage to the rolls, when rolling thicker specimens. The material, however, still showed much resistance to cold rolling, and thinner samples proved the least likely to crack under these circumstances.

Annealing

All annealing was carried out in a porcelain tube furnace, externally heated by electrical resistance windings. The temperature was controlled to $\pm 5^{\circ}\text{F}$. The tube of the furnace was sufficiently long that the annealing specimen could be pulled to cool portions of the tube for controlled rates of cooling, as desired, and still be maintained in the inert atmosphere of the furnace. Dried argon was used as the annealing atmosphere. The gas was sufficiently dry that, if the test pieces were heated and cooled in the atmosphere, no surface oxidation was observable.

Because of the small size of the specimens used for the annealing temperature investigation, and for the cold work plus anneal study ($3/4'' \times 1/4'' \times 1/4''$), the specimen was presumed to heat up to furnace temperature very quickly. Consequently, annealing times were measured

from the time of inserting the test specimens into the furnace.

Metallographic Preparation

Since air cooling was the predominant method of cooling from the annealing temperature, most specimens from the annealing temperature study and from the cold roll plus anneal investigation were covered with a light gray (presumably oxide) film on cool-down to room temperature from the annealing temperature. This film was very thin, and was easily removed by light abrasion with fine emery paper.

Most of the specimens used in the portions of the program reported upon herein were sufficiently small that they had to be mounted for metallographic polish, etch, and examination. Lucite transoptic powder and quickmount liquid plastic was used as the mounting material.

Standard metallographic polishing techniques were used for the preparation of the materials for microscopic examination and grain size measurement. This consisted of grinding and polishing on standard emory paper and final polishing on 0.3 and 0.06 alumina impregnated wheels.

It was found necessary to employ several different types of etchants and etching techniques to reveal the desired grain structures for study. The first, for essentially macro-etching characteristics, was a chemical etch consisting of one part of chromic acid and three parts of hydrochloric acid. The second was an electrolytic etch,

consisting of seven parts of methanol, four parts of glycerine, and one part of nitric acid. This etch was useful in delineating the grain structures on some of the specimens. This third, and perhaps most useful, etch was another formulation for electrolytic etching, and consisted of 33% nitric acid, 33% acetic acid, 33% water, and 1% hydrofluoric acid. This last etch seemed to be capable of bringing out the fine grain structures that many of the specimens possessed.

The etching voltages ranged from one to ten volts, while the etching times had to be varied from two to 20 seconds.

Despite the fact that all specimens polished and etched were essentially of the same composition, they showed quite widely different reactions to the etches. At present, an explanation for this difference is not apparent.

Microhardness Measurements

In order to identify the physical changes which were occurring during the annealing study, and more particularly during the study of the effects of cold work plus annealing, microhardness tests were made on specific specimens. For these determinations, the Knoop indenter, with a 300 gm. load was employed. Depending upon the scatter encountered, the number of indentations made on each specimen varied from three to ten.

Grain Size Measurement

The grain sizes were determined as a function of the various

modes of deformation and annealing. Since most of the grains encountered were not equiaxed, a volume grain size measurement was necessary. Specimens were examined in the longitudinal, long transverse, and short transverse planes. Photomicrographs were taken on the longitudinal plus short transverse plane (Figure 8), since it was in this plane that the response to deformation and annealing was most apparent. Grain size measurements were made using the line-intercept method, using at least 25 intercepts and averaging the grain size obtained for each intercept. The measurements were made in the three mutually perpendicular principal planes, obtaining an average grain dimension for each.

When they occurred (and where it was feasible), duplex grain structures were considered to be composed of material with two different grain sizes, and the average grain dimension of both the large and the small sections of the microstructure were measured individually. In those cases in which good delineation between the large and the small grain sizes was not possible, average measurements were made as a matter of course, but the results were not used in the analysis.

For those specimens which were not equiaxed in the plane of grain size measurement, the aspect ratio, or the ratio of the length of the grain to its width (L/D), was computed from the average length and width measurements. In those specimens showing duplex grain sizes, the aspect ratio of elongated grains was measured for comparison purposes. In all cases of duplex grain size materials, it was the

larger of the grains which showed elongation.

RESULTS AND DISCUSSION

As discussed earlier, the primary goals for the work reported upon herein dealt with finding the processing treatment(s) which would produce the most favorable grain size and shape in the Nicraly. To this end, the techniques which were found to be successful for the TD-Nickel and the TD-Nichrome were employed first. These consisted of sequences of cold work (by rolling) and annealing to produce recrystallization in the material.

In order to produce controlled amount of recrystallization and grain growth, work on the previous materials indicated that the best control points were the amount and direction of the cold work, along with the temperature of the final anneal. This technique was employed for the Nicraly, with the amount of reduction in thickness by rolling at room temperature, the direction of the rolling (parallel to, or at right angles with, the extrusion direction), and the temperature of the final recrystallizing anneal being the control parameters.

The high alloy content of the Nicraly rendered it intrinsically hard in the as-extruded (as-received) condition, and in this condition, it was capable of little cold working. Consequently, efforts were directed at softening it to permit significant amount of cold work. The results of this part of the study are discussed below.

The recrystallization work, i.e., the program of cold work plus

anneal to promote grain size and shape control, was rather extensive. Unfortunately, it was necessary to conduct most of it on the material from the 101 Powder Batch, rather than on the material from the preferred 204B Powder Batch (as discussed earlier). The results of this study form the major portion of the work considered in this report. It is discussed below in appropriate divisions, i.e., it is considered in terms of each of the major parameters.

Characterization of the As-Extruded Material

The Nicraly used in this study was reported to have been supplied in what is nearly an equilibrium state. The processing techniques used to produce the extrusions supplied is reported to have yielded a material with a very low stored strain-energy state. Annealing studies with this "as-extruded" or "as-received" material tend to confirm this condition of the material.

The microstructure of the preferred 204B material, as supplied as extrusions, is shown in Figure 1(d). In general, the material has a relatively small grain size, and a rather large grain aspect ratio. The average grain size value was 0.0228mm., and the aspect ratio varied from 8:1 to 30:1. The basic grain size and structure of the 101 and 204B materials are identical in grain size and shape as shown in Figure 1(d).

As regards effects which might stem from the two different powder batches from which the extrusions were made, perhaps the greatest

manifestation of any substantial difference might be seen in the microstructures of the two materials. The material made from Powder Batch 101 showed small spherical microconstituents which were not present in the material made from Powder Batch 204B. The supplier of the materials indicated that Powder Batch 101 was a transition formulation, and while it had the correct overall chemical composition, it would not necessarily have the preferred properties. The presence of the spurious microconstituent would seem to confirm this surmise.

The form and distribution of the spurious phase in the 101 material suggested that it might represent some kind of chemical inhomogeneity. This suspicion seemed to be supported by the significant difference in the hardness of the as-extruded materials from two powder batches, as shown below*:

Powder Batch 101	462 Knoop
Powder Batch 204B	514 Knoop

While the above hardness difference is not exceptionally large, it is still beyond the range of normal scatter of the measurement. If, in fact, the spurious phase in the 101 material were a chemical segregate of the element(s) which normally contribute to the solid solution hardness of the matrix material in the ODS alloy, its concentration into a second discrete phase might produce the hardness difference noted above.

Again, it should be recognized that the 101 material was only a

*Hardness data for all of the material conditions developed in this study are shown in Table 1.

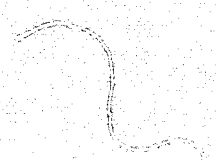
substitute material which was used in this study only until material of the preferred manufacturing process could be procured. Consequently, it was not deemed advisable to expend the resources necessary to resolve the nature of the extraordinary phase present in the 101 material.

Preliminary Annealing Study

As noted above, the extruded material was annealed before attempting to cold work it by rolling, in an effort to maximize its homogeneity and its softness. The annealing treatment selected was based upon the results of a preliminary annealing study.

Information received from the material supplied indicated that maximum softening could be expected from the highest annealing temperatures that were practical, 2400°F to 2490°F. (Incipient fusion occurs at 2500°F for this material.)

Consideration was given to the rate of cool from the annealing temperature in this study. The alloy composition is in the range for the formation of the gamma prime phase, at least theoretically. While the study of materials with this second phase in the matrix is an interesting adjunct to the present program, it was felt that the study of material with the single-phase FCC matrix should precede it. Consequently, the rate of cool was investigated, since the sluggishness of the system suggests that gamma prime will form only on slow cooling. Conversely, rapid rates of cool should optimize softening.



The flow charts which describe the preliminary annealing study are shown as Table 2 for the material from Powder Batch 101 and as Table 3 for the material from Powder Batch 204B. The work on the former material preceded that on the latter, and hence the results of the 101 material were used to set the program for the 204B material.

The annealing study for the 101 material included two cooling rates . . . a slow furnace cool, and an air cool. A faster water quench was added for the 204B material in an effort to maximize the softening of material further before rolling. In order to minimize the possibility of overlooking any unusual annealing effects, a fuller annealing temperature range (1600 to 2450°F) was used on the 204B material as compared with that used for the 101 material; also one longer annealing time (24 hours). This latter longer time was added for the 204B material to obtain some qualitative measure of the rate of growth of the large grains.

The effect of cooling rate from the 2450°F (1343°C) annealing temperature on the microstructure of the 101 material is shown in Figure 1(a-c) and in Figure 2. Little difference can be seen as a result of the different rates of cool, in terms of general grain conformation or microconstituents. Quantitatively, some small differences were noted, however. The comparison of the grain size and aspect ratios is listed below.

<u>Rate of cool</u>	<u>Avg. Grain Dim.</u> <u>mm</u>	<u>Aspect Ratio</u>
Air cool	0.874	30:1
Furnace cool	0.9804	14:1

When the 101 material was annealed at the highest possible temperature, 2490°F (1366°C), and furnace cooled, there was some increase in the grain size over that in the material which was annealed at 2450°F (1343°C) and furnace cooled. The average grain diameter was 1.668 mm, and the aspect ratio was 21:1.

The annealing treatments produced substantial changes in hardness from that of the as-extruded material, as summarized below:

101 MATERIAL

<u>Annealing Treatment</u>	<u>Knoop Microhardness</u>
As-extruded	462
Anneal one hour at 2450°F, Air cool	255
Anneal one hour at 2450°F, Slow cool	332
Anneal one hour at 2490°F, Air cool	313

Rather interestingly, the treatment involving an air cool from the 2450°F annealing temperature produced the greatest softening of the extruded material. In the interests of pursuing the main thrust of the study, no attempt was made to rationalize the fact that the treatment involving furnace cool from 2450°F and the 2490°F annealing

temperature produced less softening than that utilizing an air cool from the 2450°F annealing temperature.

Extension of the preliminary annealing study to the 204B material showed that it responded to the annealing in the same general manner as did the 101 material. Recrystallization was complete at all temperatures above at least 2000°F; rather large elongated grains were formed; considerable softening occurred. The largest final grain size was obtained at the highest annealing temperature used, 2450°F. The grain size and shape for the 2450°F annealing schedules studied are listed below:

<u>204B MATERIAL</u>		
<u>Rate of Cool from 2450°F</u>	<u>Avg. Grain Dim. mm</u>	<u>Aspect Ratio</u>
Air Cool	0.922	10:1
Water Quench	2.324	17:1

The maximum softening produced as the result of the study was less than that obtained for the 101 material, but was still substantial. As was expected, the maximum softening was obtained with the highest annealing temperature employed, 2450°F. The degree of softening obtained on the 204B material is summarized below:

204B MATERIAL

<u>Material Condition</u>	<u>Knoop Microhardness</u>
As-Extruded	514
Anneal one hour 2450°F Air Cool	352
Anneal one hour 2450°F Water Quench	309
Anneal 24 hours 2450°F Air Cool	308

The data shown above would indicate some variability in the material. Because of the small size of the annealing study specimens, it seems unlikely that there should be a difference in the actual cooling rate between air cool and water quench sufficient to produce the 43 points of hardness difference between the two specimens annealed at 2450°F. This same kind of variability is shown on the previous tabulation for the 204B material, which shows a larger grain size and aspect ratio for the water quenched specimen than for the air cooled specimen, both of which were annealed identically. It is highly unlikely that the cooling rate could produce such an effect.

Despite the fact that the water quench from the annealing temperature produced material with a large (or largest) grain size, and a high aspect ratio, its use could not be considered in establishing the final annealing schedule for the preliminary anneal, because the water quenched specimens showed quench cracks. While these cracks could be tolerated in test specimens being used only for hardness and

and microstructure study, they could not be tolerated in creep and rupture test pieces.

On the basis of the above data, the decision was made to use the one hour anneal at 2450°F (1343°C) with air cool as the standard pre-rolling annealing treatment. The rationale behind this decision is the following:

1. Annealing temperature: 2450°F (1343°F) is the highest temperature at which an absence of incipient fusion can be assured, and at which the increase of grain size with increasing temperature can be exploited.
2. Annealing time: one hour appeared to produce as large a grain size at the longer annealing time (24 hours), with a minimization of danger of specimen damage because of abnormal temperature fluctuations.
3. Cooling rate: air cooling produced the largest grain size of any cooling rate, without danger of cracking (as was found in the 101 material which was water quenched), and without danger of forming the gamma prime phase (which has been reported on occasion for material of this composition with controlled slow cooling).

It might be noted that the water quenched specimen of the 204B material annealed for one hour showed a larger grain size than any of the other specimens of the study. However, the difference was only

a factor of about 2.5 (in comparison with the air cooled specimens), and differences of orders of magnitude are usually necessary to produce marked effects of high temperature properties. Further, as regards the furnace cool, it should be noted that no gamma prime microconstitutents were noted in the optical microscope examination of the specimens which had the very slow cooling rate. However, this does not guarantee the absence of small sub-microscopic (optical) particles, and since there was little to be gained by using the slow cool, the decision was made to avoid its use.

Cold Roll plus Anneal Study

As noted earlier, the main thrust of the attempts to achieve grain size and shape control employed the "cold work plus recrystallizing anneal" principle. After the standardization anneal of the as-extruded material, both the 101 and 204B material were cold rolled in the longitudinal (extrusion) direction up to the maximum of which they were capable (determined by material breakage). Several intermediate reductions were then chosen as "standard" amounts of cold work, and recrystallizing anneals were given to material cold worked by these amounts. The Flow Charts shown in Tables 4 and 5 summarize the processing schedules, while Figure 8 shows the relevant directions of rolling.

In addition, the 204B material was cross-rolled, i.e., rolled at right angles to the extrusion direction, up to the maximum possible. This maximum was again determined by material breakage. As for the

material which was rolled in the extrusion direction, intermediate reductions were chosen as "standards" for the cross rolled material, with annealing studies being conducted on them. The Flow Chart in Table 6 summarizes these processing schedules.

Work Hardening Characterization

The work hardening characteristics of the materials used in the study are shown in Figure 9 for the 101 material. The 204B material showed a similar response to the cold curve. Both showed the classical hardening response to longitudinal cold rolling, with large increases in hardness resulting from initial, small amount of cold work, small increments resulting from intermediate amounts of cold work, and again large increments resulting from large amounts of cold work. Average microhardness data for the cold worked materials are summarized below:

<u>101 Material</u>	
<u>Material Condition</u>	<u>Knoop Microhardness</u>
annealed 1 hour 2450°F air cooled	255
preannealed 1 hour 2450°F/longitudinally rolled 25%	426
preannealed 1 hour 2450°F/longitudinally rolled 38%	475
preannealed 1 hour 2450°F/longitudinally rolled 50%	458
preannealed 1 hour 2450°F/longitudinally rolled 73%	480
<u>204B Material</u>	
annealed 1 hour 2450°F air cooled	352
preannealed 1 hour 2450°F/longitudinally rolled 20%	517
preannealed 1 hour 2450°F/longitudinally rolled 25%	381
preannealed 1 hour 2450°F/longitudinally rolled 30%	460

The trend of the change in hardness with increasing amounts of cold work is much better defined for the 101 material (see above listing and Figure 9) than it is for the 204B material, because the former was capable of much more cold work than the latter (73% versus 30%). For both materials, however, the hardness of the blanks which were cold rolled 20% was somewhat higher than might have been expected on the basis of the balance of the data and the trend curve. No attempts were made to rationalize this (apparent) abnormality. If work presently underway indicates that there is some special reason for utilizing the 20% reduction as one of the preferred cold working steps, efforts will be made to resolve the apparent irregularity.

While some attempts were made to cross roll the 204B material, it fractured after only very small amounts of cold work for both heavy and light reductions per rolling pass. Since the cold work which was given to the material on the cross rolling was small, it was felt that most of the reduction in thickness that was achieved was probably concentrated near the rolling surfaces of the material. Hence, no attempts were made to develop a hardness versus amount of cold work relation. If the material were, in fact, capable of high reduction by cross rolling, the trend curve showing the work hardening characteristics of the material would be expected to resemble that shown in Figure 9.

The microstructures of the cold worked materials were examined in detail, but showed only the expected results of the cold working. The

grain size (volume) was essentially constant, but the aspect ratio increased in the rolling direction.

On the basis of the work hardening curves, the intermediate reductions chosen for annealing response studies were 25%, 38%, 50% and 73% for the 101 material which had been rolled in the extrusion direction, 15%, 20%, 25% and 30% for the 204B material which had been cold worked by rolling in the extrusion direction, and 12% and 15% for the cross rolled 204B material. Referring to Figure 9, these cold reductions correspond to small, intermediate, and large amounts of cold work.

The most detailed of the annealing studies was conducted on the 101 material which had received 38% cold reduction. Referring to Figure 9, the as-rolled hardness (and therefore presumably the stored strained energy) for this material was about the same as that with 25% cold work. However, the former reduction was chosen over the latter because it was felt that the cold work would have been more uniformly distributed throughout the thickness of the piece when the cold reduction was greater for the same hardness level. Further, as a matter of interest, this 38% reduction corresponded to one-half of the maximum of which the material was capable, and was one which could be employed commercially with convenience.

Cold Work Plus Anneal Response of the 101 Material,
Longitudinally Rolled

The response of the 101 material cold rolled various amounts and

annealed at temperatures ranging from 700°F to 2450°F was manifest in the changes in microstructure. The various features of the microstructural changes are shown in Figure 3(a-d) for the material with 25% cold work, and are summarized below:

101 MATERIAL - 25% REDUCTION, LONG. ROLLING

ANNEALING TEMPERATURE °F	GRAIN CHARACTERISTICS		
	AVG. GRAIN DIMENSION (mm)	ASPECT RATIO	UNIFORMITY
R.T.	0.188	11:1	Good
700	0.345	16:1	Good
1000	0.579	13:1	Good
1560	1.422	24:1	Good
1900	0.914	22:1	Good
2450	0.102	29:1	Duplex

From the above listing, it is quite clear that the largest grain size was that obtained after the anneal at 1560°F. It would appear that the material annealed at 700°F and 1000°F did not recrystallize. The reduction in grain size after annealing at 1900°F and 2450°F appears to be the result of the activation of increasing numbers of nucleation sites as the temperature of the anneal is increased.

The relevant microstructures of the 101 material which had been cold worked 25% by rolling and annealed at various temperatures are shown in Figure 3. In the as-rolled condition, the elongated grains

showed distinct slip bands or traces, Figure 3(a). Annealing at the low temperatures produced no recrystallization, Figure 3(b). However, annealing at the 2450°F (1343°C) temperature caused recrystallization, with a resulting duplex grain structure, Figure 3(c). The fine-grain component of the duplex structure seemed to have formed along slip bands in the elongated grains which resulted from the cold working.

As noted earlier, the 101 material cold rolled 38% was selected for the intensive annealing study. The results of annealing this material at temperatures between 1000°F (538°C) and 2450°F (1343°C) on the grain size of the material are summarized in the tabulation below, while the effects of annealing temperature on the hardness of the material are shown in Figure 10. The various relevant microstructures for the materials of the study are shown in Figure 4.

101 MATERIAL - 38% REDUCTION, LONG. ROLLING

ANNEALING TEMPERATURE °F	GRAIN CHARACTERISTICS		
	AVG. GRAIN DIMENSION (mm)	ASPECT RATIO	UNIFORMITY
R.T.	1.4199	36:1	Good
1000	0.3861	14:1	Good
1400	1.219	39:1	Good
1500	3.241	53:1	Good
1000	2.4562	155:1	Some Duplex
1700	1.661	39:1	50% Duplex
1900	0.0381	41:1	Duplex
2000	0.0381	38:1	Duplex
2200	0.4978	29:1	Duplex
2300	0.0432	33:1	Duplex
2400	0.0419	44:1	Duplex
2450	0.0386	26:1	Duplex

To summarize the overall findings of this exhaustive annealing study, the collective data seem to indicate that no recrystallization of the material had occurred at annealing temperatures below 1500°F (816°C), but had begun to some limited extent at 1600°F (871°C). Further, the recrystallized material took the form of small, equiaxed grains, producing the duplex microstructure. As the annealing temperature was increased progressively from 1600°F (871°C), the amount of recrystallized material increased, but the grain size of the recrystallized material remained essentially constant. Even at the highest annealing temperature used, 2450°F (1343°C), some of the original cold worked grains remained. A few words of explanation of the above findings might be in order.

The as-rolled material had the expected elongated grain form, Figure 4(a), with the grain volume being unchanged. This resulted in an aspect ratio which was greater than that of the material which had a smaller amount of cold work. (The aspect ratio for the material cold rolled 25% was 11:1, while that cold rolled 38% was about 36:1) As for the material with a lesser amount of cold work, strong slip bands were present in the material.

As the temperature of the anneal after the cold rolling was increased up to 1500°F (816°C), there were no visible changes in the microstructure over that of the as-rolled material, as is evident from a comparison of Figure 4(a) with Figures 4(b), 4(c), and 4(d). The tabulation of the grain dimensions and shape, shown above, indicate some scatter in the average grain dimension and the aspect ratio,

but this is probably due to the basic nature of the material. In essence, then, the grain size and shape had not changed.

Annealing at temperatures up to 1500°F (816°C), while producing no sign of shape changes, did effect a drop in hardness, Figure 10. A more quantitative indication of the drop in hardness is evident in the listing below:

101 MATERIAL - 38% REDUCTION, LONG. ROLLING

<u>Annealing Temperature °F</u>	<u>Knoop Microhardness</u>	<u>Annealing Temperature °F</u>	<u>Knoop Microhardness</u>
R.T.	475	1600	411
700	427	1700	363
800	449	1800	325
900	443	1900	353
1000	422	2000	339
1100	473	2100	346
1200	401	2200	311
1300	468	2300	327
1400	448	2400	305
1500	369	2450	301

There appeared to be a drop of some 75 Knoop hardness numbers from the as-rolled hardness when the material was annealed at 1500°F (816°C). On the basis of the balance of the data, it would appear that this is the classical relaxation which is often the predecessor of recrystal-

lization process in cold worked materials.

When the cold rolled material was annealed at 1600°F (871°C), small equiaxed grains were formed, Figure 4(e), although their presence is difficult to detect in the photomicrograph. In general, the amount of the small grain component of the duplex structure increased as the annealing temperature was increased beyond 1600°F (871°C), Figures 4(f) through 4(l), with the last photomicrograph of the series being that for material which was annealed at the highest practical temperature, 2450°F (1343°C). The elongated grain component of the duplex structures shown in Figures 4(f) through 4(l), appear to be the original elongated, cold worked grains, because they have the slip bands characteristic of the cold worked material, Figures 4(g) and 4(j).

The increase in the amount of the recrystallized material is manifest in the drop in hardness as the annealing temperature was increased from 1600°F (871°C) to 2450°F (1343°C), with the drop following the classical sigmoidal shape, characteristic of recrystallization curves, Figure 10.

The aspect ratio of the unrecrystallized grains, as shown in the tabulation of these data for the 38% cold rolled material, was essentially constant, with the range of values (10:1 to 100:1) being characteristic scatter in the material.

The average grain dimension shown in the tabular listing of the data for the material with 38% cold work reflects the increasing

amount of the fine recrystallized component of the duplex structures which were present after annealing at temperatures above 1600°F (871°C).

On the basis of this study, it would appear that the elongated grains produced by cold rolling 38% are quite stable, or resistant to recrystallization, and that when recrystallization does occur, the new grains are fine, equiaxed, and resistant to growth.

The results of the annealing study on the material which had been cold worked 50% by longitudinal rolling followed the trend set by the extensive study of the material with 38% cold work. The results of the microstructural changes brought about by the annealing are summarized below:

101 MATERIAL - 50% REDUCTION, LONG. ROLLING

ANNEALING TEMPERATURE °F	GRAIN CHARACTERISTICS		
	AVG. GRAIN DIMENSION (mm)	ASPECT RATIO	UNIFORMITY
R.T.	2.471	37:1	Heavy Slip Bands
1000	0.813	17:1	Slip Bands
1600	0.0085	-	Duplex
2000	0.016	-	Equiaxed Grains
2450	0.023	-	Equiaxed Grains

The as-rolled material showed exceptionally heavy slip banding, Figure 5(a), and highly elongated grains. On annealing at a tempera-

ture below recrystallization, 1000°F (538°C), it appeared that some of the slip lines had relaxed, but the material was still heavily slip-banded. While the aspect ratio is shown to be less than that of the as-rolled material, the difference is probably statistical scatter.

On annealing at 1600°F (871°C), a duplex grain structure formed, with the equiaxed grains being very small. A portion of the fine-grained component of the material is shown in Figure 5(b) (note higher magnification than in previous photomicrographs).

On annealing at 2000°F (1093°C) and 2450°F (1343°C) recrystallization occurred quite extensively, shown in Figures 5(c) and 5(d). Note again the high magnifications of the photomicrographs that were necessary to delineate the grain forms. Rather interestingly, there were still some semblances of the original cold worked grains at the higher annealing temperatures, Figure 5(c). The most significant point, however, is perhaps the extremely small size of the recrystallized grains, and their apparent resistance to growth (the grain size of the material annealed at 2000°F was essentially the same as that annealed at 2450°F).

For the 101 material cold worked the maximum amount (73% reduction in thickness), the annealing response followed the same trend as that outlined above for the material with 50% cold work, but producing even finer grains in the recrystallized material. The summary of the effects of the annealing study on this heavily cold worked mater-

ial are summarized below:

101 MATERIAL - 73% REDUCTION, LONG. ROLLING

ANNEALING TEMPERATURE °F	GRAIN CHARACTERISTICS		
	AVG. GRAIN DIMENSION (mm)	ASPECT RATIO	UNIFORMITY
R.T.	1.143	27:1	Slip Bands
1000	1.397	36:1	Slip Bands
1600	0.457	-	Nearly Equiaxed
2000	0.0279	-	Equiaxed
2450	0.0096	-	Equiaxed

The highly elongated grain structure of the as-rolled material is shown in Figure 6(a), which also portrays the heavy slip-banding present in the rolled material. Annealing at 1000°F (538°C) produced no apparent change in the microstructure, Figure 6(b). However, raising the annealing temperature to 2000°F (1093°C) produced nearly complete recrystallization to a fine equiaxed grain structure, Figures 6(c) and 6(d). A further increase in annealing temperature to 2450°F (1343°C) also produced complete recrystallization, Figures 6(e) and 6(f), and again with exceedingly fine grains. In this latter case, however, there did appear to be some very small amount of growth of the recrystallized grains.

In the examination of the microstructure of the material cold rolled 73% and annealed at 2450°F (1343°C), a rather unusual effect was noted in the grain boundary. There appeared to be a veinous net-

work of a new phase present in the boundaries. However, it became distinct only at the very high magnifications (1000 and 2000 times), Figures 6(g) and 6(h). At first this was thought to be an etching artifact; however, the network persisted through several repolish and re-etching cycles, and with the use of several different types of etches. Consequently, it might well be a second phase in the matrix, although its presence at grain boundaries only cannot be rationalized.

Cold Work Plus Anneal Response of the 204B Material,
Longitudinally Rolled

To compare results of the 101 with the 204B material, sample rolling reductions followed the processing schedule as shown in Table 5. The reactions of the 101 material to cold rolling and annealing were again found in the 204B material. The duplex grain structure appeared at 2000°F in all the rolling reductions. The equiaxed grain structures appeared at the 2450°F annealing temperature as in the 101 material.

Cold Work Plus Anneal Response of the 204B Material,
Cross-Rolled

The lack of success in attaining large recrystallized grains with the 101 material, together with the fact that the 101 and the 204B materials were generically the same, prompted the elimination of the longitudinal cold rolling plus annealing study of the 204B material, when it became available. Consequently, the 204B material was subjected then to a cross-roll plus anneal study.

Unfortunately, the limited transverse ductility of the 204B material placed severe restrictions on the amount of cold work that could be put into the material by cross-rolling at ambient temperature. It was found that the cross-rolling blanks cracked when the total reduction reached approximately 15%. The chart of the processing operation is shown in Table 6.

Grain size results for the material rolled 12% and annealed at 2450°F are given below:

<u>204B MATERIAL</u>			
12% Reduction Transverse Rolling			
<u>GRAIN CHARACTERISTICS</u>			
<u>ANNEALING TEMPERATURE °F</u>	<u>AVG. GRAIN DIMENSION (mm)</u>	<u>ASPECT RATIO</u>	<u>UNIFORMITY</u>
R.T.	2.692	33:1	Good
2450	2.134	16:1	Duplex

The microstructures of the material rolled to 12% and 15% reductions, and annealed at 2450°F only, to check for the small equiaxed structures, are shown in Figure 7. The cross rolled material had larger grain dimension, however, the duplex grain structure was again evident to a smaller extent in the annealed specimen, which accounted for the smaller grain size shown in the table, after anneal at 2450°F.

CONCLUSIONS

On the basis of the work completed to date on the nickel base alloy, "nicrally", an oxide dispersion strengthened alloy made from powder blends containing 16% chromium, 4% aluminum, 2-3% yttria, balance nickel, much has been learned about the material's response to annealing, to cold working by rolling, and to combinations of cold work and annealing, in terms of its final grain size and grain shape.

Though the material has many generic similarities to predecessor ODS alloys, it is vastly different in this response to thermal and/or mechano-thermal processing. Specifically, it is not amenable to the control of its grain size and shape by thermo-mechanical processing in the same way that TD-Nickel and TD-Nichrome were. It does not undergo rapid recrystallization and immediate abnormal grain growth on annealing after substantial cold working as did TD-Nickel. It does not lend itself to grain size control by classical nucleation and grain growth mechanisms, as did TD-Nichrome.

On the other hand, the Nicrally is quite amenable to grain size control on annealing the as-extruded material at temperatures approaching the melting range for the alloy. Favorably large grains, with relatively high aspect ratios can be produced by such anneals. Further, the aspect ratio of the grains can be increased by unidirectional cold working at ambient temperatures, since the material is sufficiently ductile to permit sizeable mechanical working deforma-

tions. In addition, the highly elongated grains produced by the uni-directional working were relatively stable as regards recrystallization on heating to moderately high temperatures.

The Nicrally appears to be quite sluggish in its nucleation of new recrystallized grains on annealing after cold working. These new recrystallized grains always seem to be fine and equiaxed. The Nicrally is further sluggish in the growth of newly formed recrystallized grains which result from annealing the cold worked alloy.

On the basis of the (still incomplete) study of the methods of grain size and shape control of the Nicrally, the most promising processing treatment for producing large grain sizes would seem to be a high temperature anneal of the as-extruded material. It is quite possible that some control of this phenomenon can be attained by control of the extrusion parameters.

FUTURE WORK

Work planned for the immediate future includes completion of the attempts to control grain size and shape of the Nicrally through thermo-mechanical processing. Specifically, the rapid increase in hardness with small amounts of cold work by rolling suggests that the material might be amenable to grain size control by the use of the (small) critical reductions to promote abnormal grain growth on subsequent high temperature annealing. This work will involve the rolling of thinner blanks than those used in the work to date, to insure through-working of the material as the low reductions in

thickness contemplated.

In addition, the thermo-mechanical processing study will be completed with an effort to exploit the use of both longitudinal and cross rolling in combination. The presence of heavy slip banding in the cold worked material studied to date suggests that texturing of the material may have an influence on the sluggishness of the response to the material to annealing recrystallization after cold work. Combinations of longitudinal and cross rolling might modify such texturing effects.

Upon completion of the items above, the entire collection of data will be assessed in conjunction with the relevant personnel at NASA (Lewis) to choose the processing schedules which produce the most favorable grain sizes and configurations. Material will then be processed to produce these grain sizes, and it will serve as the material upon which the high temperature testing will be performed.

The high temperature testing will include tensile tests at elevated temperatures, and creep and rupture testing at the same temperatures. The results of these tests will be related to the grain size, grain shape, and other material characterization values for the purposes of optimizing the high temperature performance of the material.

Data will also be collected to assess the activation energies of the high temperature deformation mechanisms. Electron microscopy will be employed to augment the study of the relevant roles of the

several deformation mechanisms which might be active at the high temperatures. Finally, collective data will be used to check the theoretical model previously developed for the mechanism of strengthening in ODS alloys. Depending upon the "fit" of the data to the model, some modification of the theoretical model might have to be made to make as nearly "universal" as possible.

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TABLE 1

MICROHARDNESS DATA FOR VARIOUS MATERIAL CONDITIONS

<u>Material Condition</u>	<u>Knoop Microhardness*</u>
<u>101 Material</u>	
as-received (as-extruded)	462.1
annealed 1 hour 2450°F air cooled	255.3
annealed 1 hour 2450°F furnace cooled	332.1
annealed 1 hour 2490°F furnace cooled	313.7
preannealed 1 hour 2450°F/longitudinally rolled 25%	426.2
preannealed 1 hour 2450°F/longitudinally rolled 38%	474.7
preannealed 1 hour 2450°F/longitudinally rolled 50%	458.4
preannealed 1 hour 2450°F/longitudinally rolled 73%	479.8
<u>204B Material</u>	
as-received (as-extruded)	513.5
annealed 1 hour 2450°F air cooled	352.0
annealed 1 hour 2450°F water quenched	309.2
annealed 24 hours 2450°F air cooled	307.8
preannealed 1 hour 2450°F/longitudinally rolled 20%	517.0
preannealed 1 hour 2450°F/longitudinally rolled 25%	381.2
preannealed 1 hour 2450°F/longitudinally rolled 30%	459.5

*300 gram load

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TABLE 2

PRELIMINARY ANNEALING STUDY

(Nicraly Powder Batch 101)

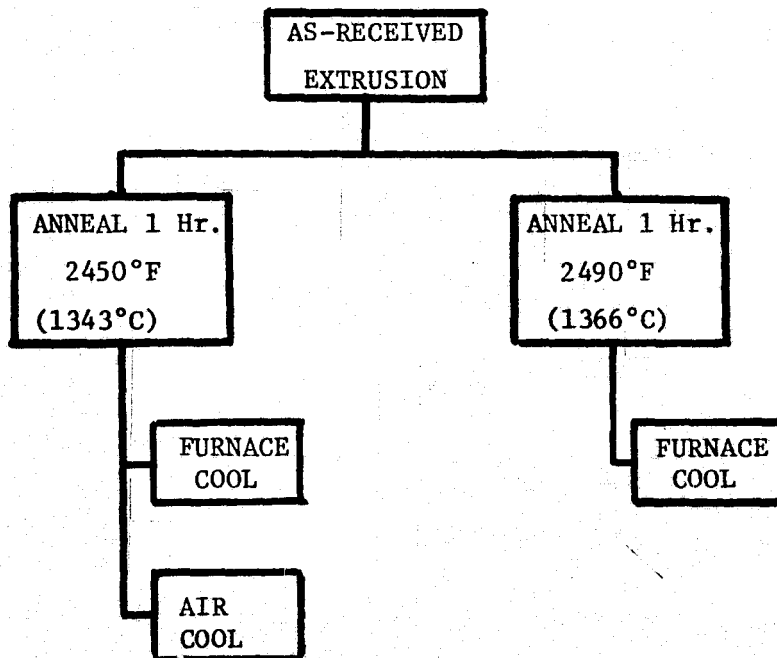


TABLE 3

PRELIMINARY ANNEALING STUDY

(Nicaly Powder Batch 204B)

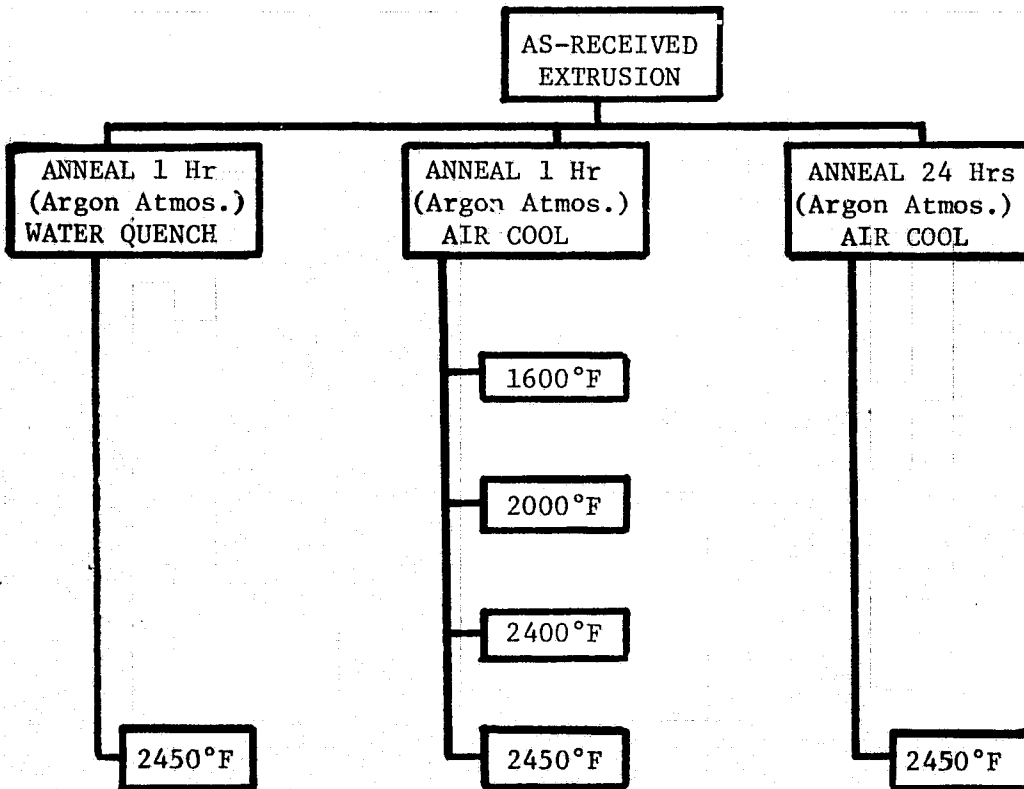
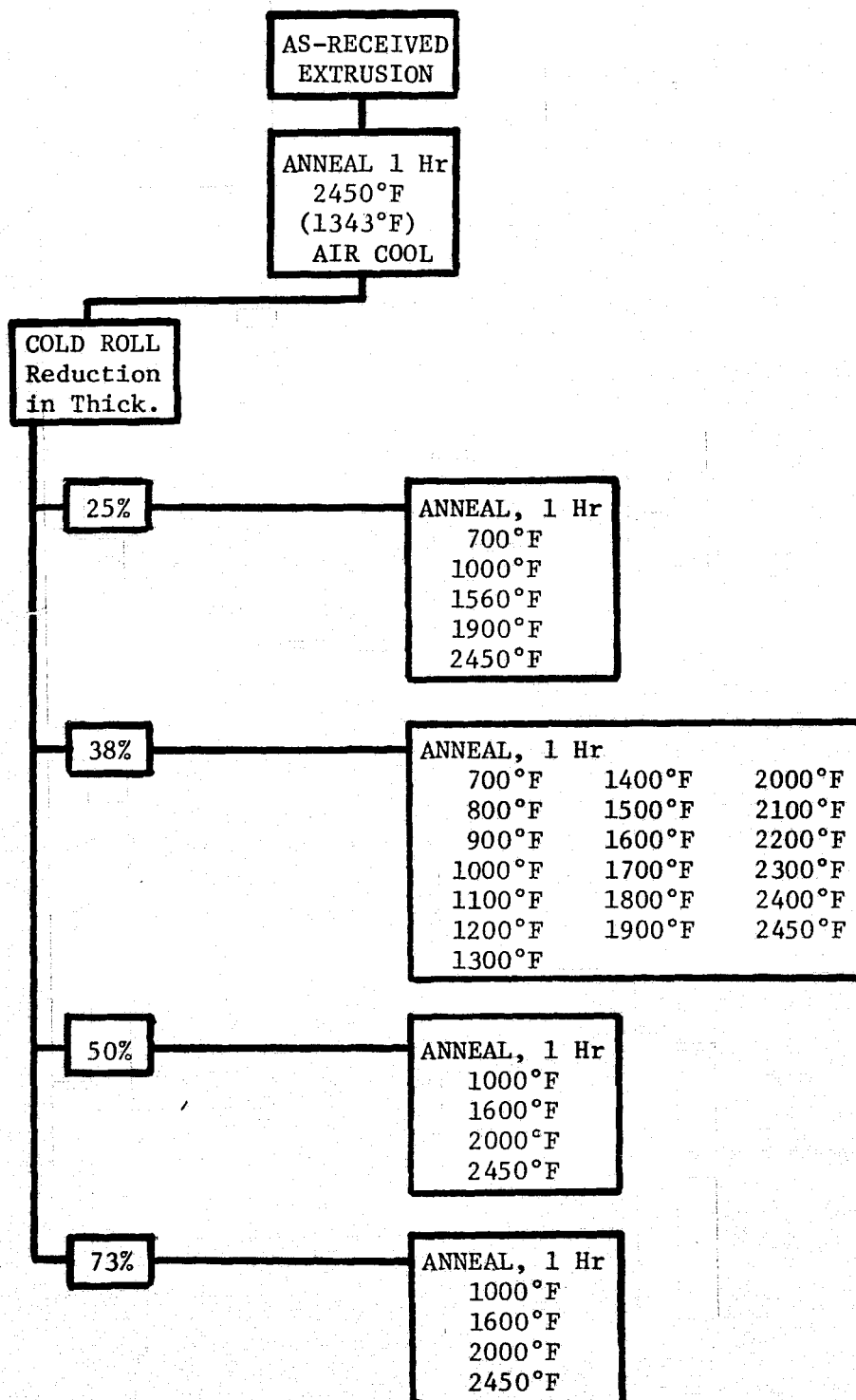


TABLE 4

COLD ROLL PLUS ANNEAL STUDY
 NICRALY POWDER BATCH 101
 ROLLED IN LONGITUDINAL DIRECTION

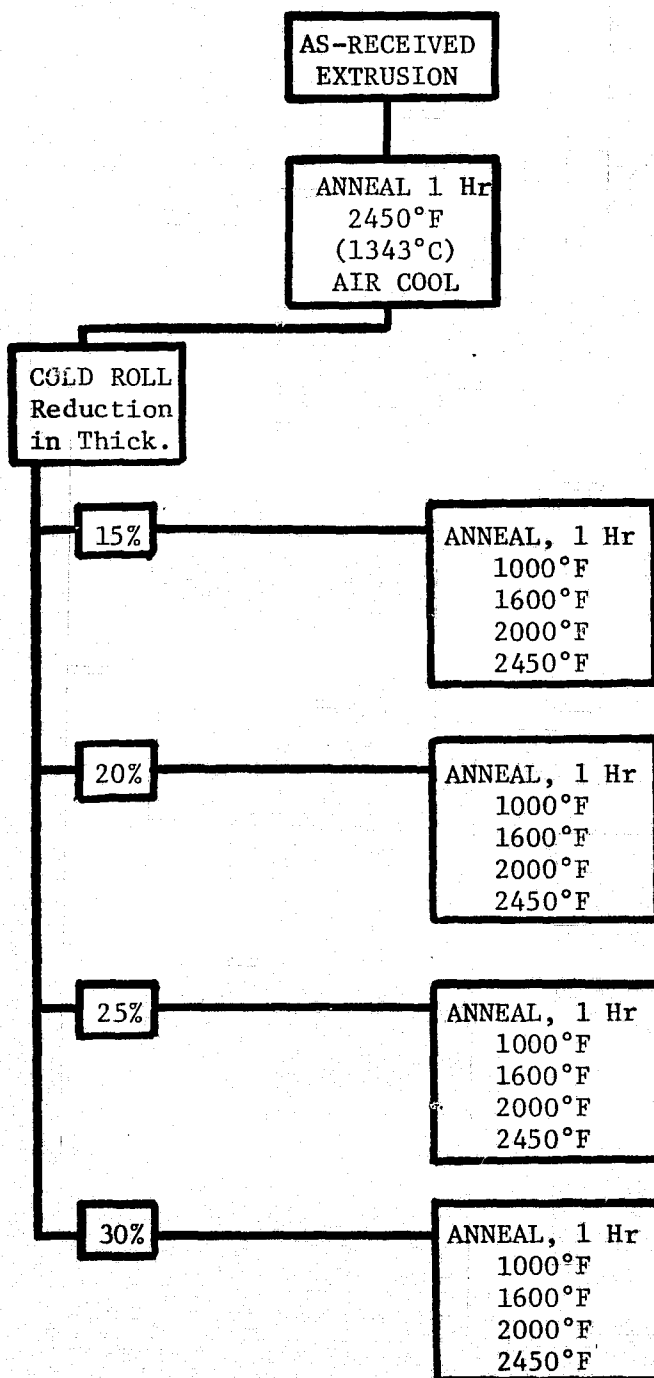


Note: All material air cooled from final anneal

TABLE 5

COLD WORK PLUS ANNEAL STUDY

NICRALY POWDER BATCH 204B
ROLLED IN LONGITUDINAL DIRECTION

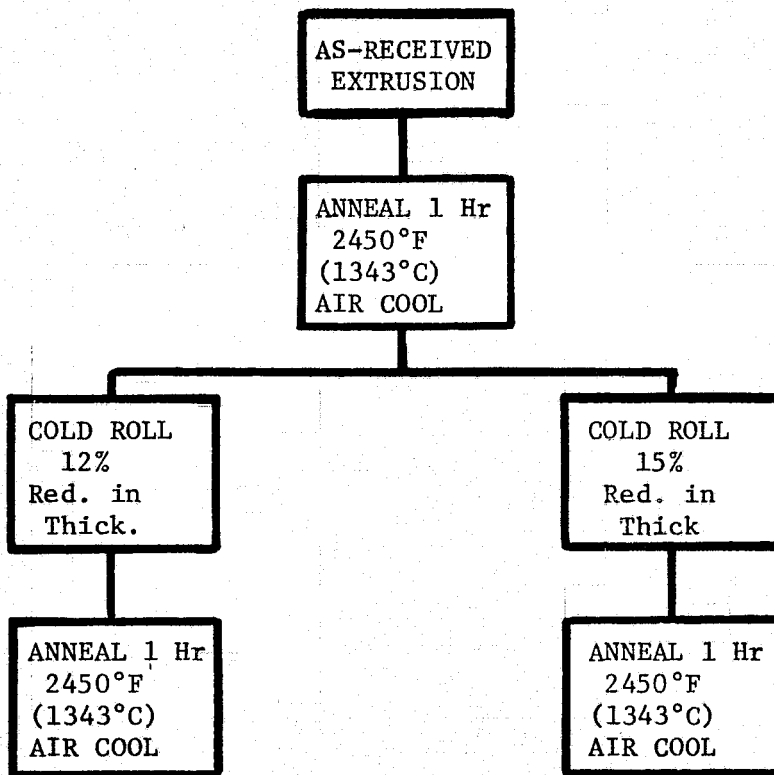


Note: All material air cooled from final anneal.

TABLE 6

COLD WORK PLUS ANNEAL STUDY

NICRALY POWDER BATCH 204B
ROLLED IN TRANSVERSE DIRECTION



1(a)
100X



1(b)
75X

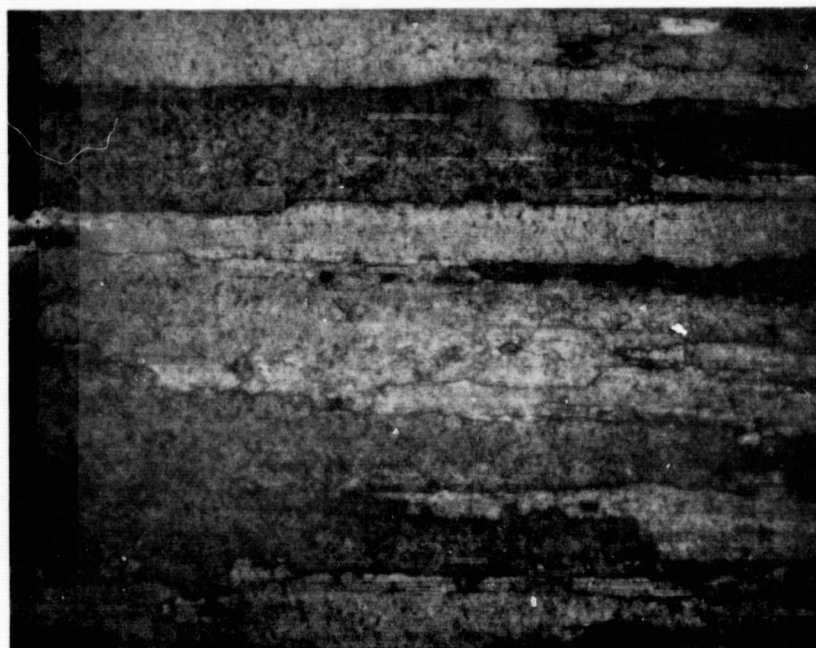
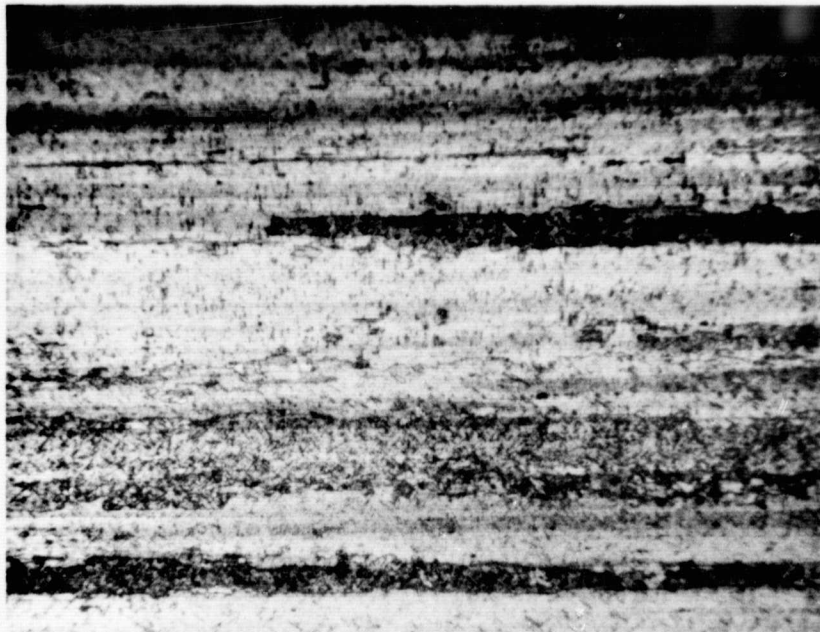


FIGURE 1: Preliminary annealing study, longitudinal and short
transverse plane shown
1(a) material 101, after annealing 1 hour at 1343°C(2450°F), slow
furnace cool
1(b) material 101, annealed 1 hour at 1366°C(2490°F), slow furnace
cool

1(c)
100X

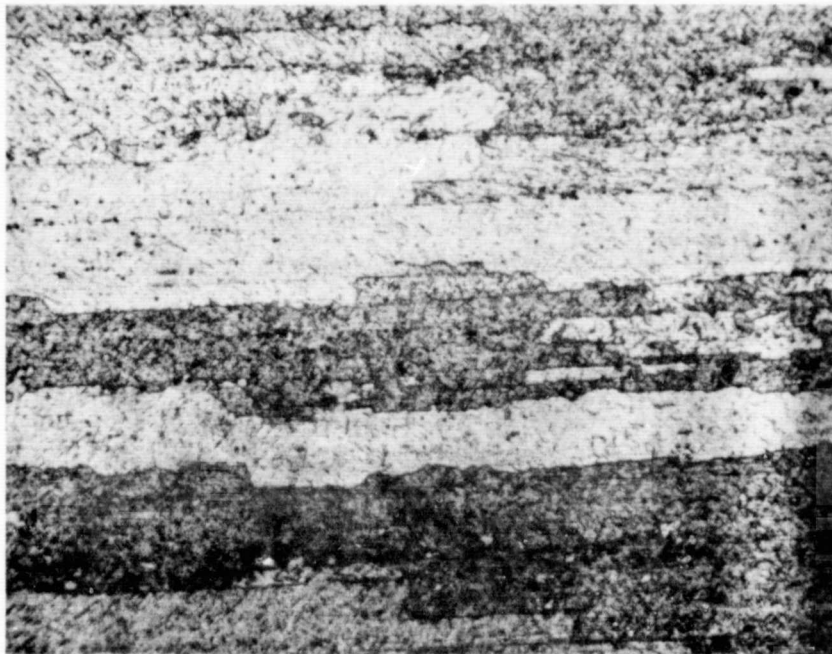


1(d)
100X



1(c) material 101, annealed 1 hour at 1343°C(2450°F), air cool, longitudinal and short transverse plane
1(d) material 204B, as received, no anneal, longitudinal and short transverse plane

1(e)
100X

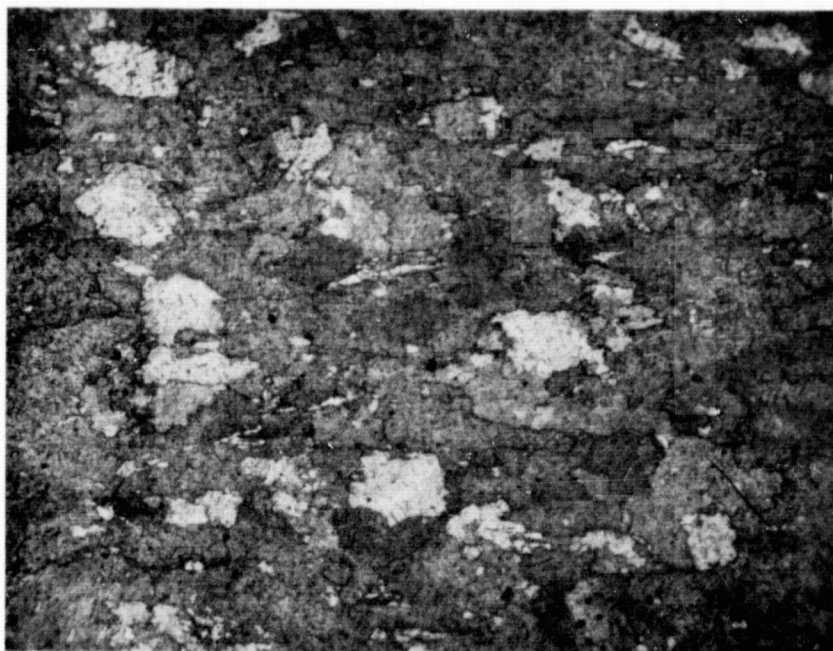


1(f)
100X



1(e) material 204B, after annealing 1 hour at 1343°C(2450°F), air cool, longitudinal and short transverse plane
1(f) material 204B, annealed 1 hour at 1343°C(2450°F), water quenched, longitudinal and short transverse plane

2(a)
100X



2(b)
200X

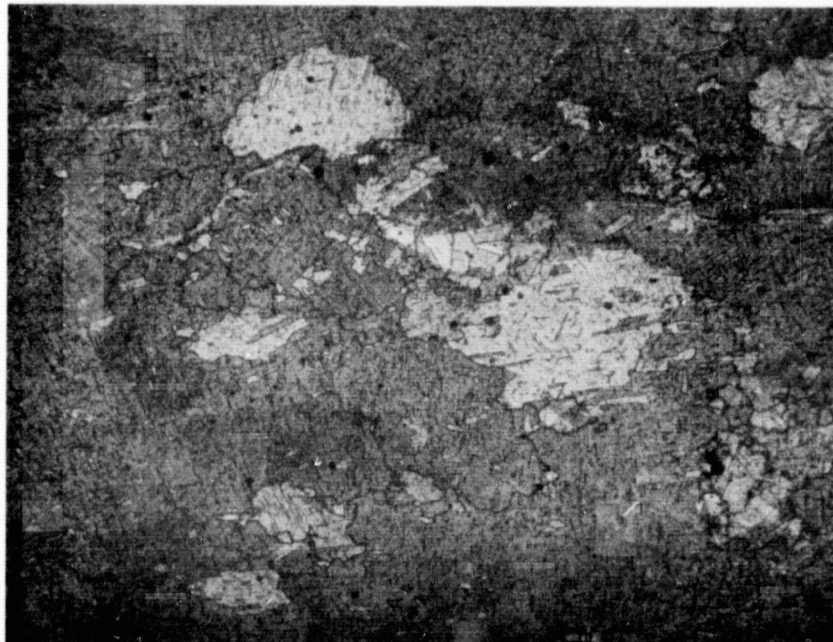


FIGURE 2: Preliminary annealing study, thickness plane shown
(a,b) material 101, annealed 1 hour 1343°C (2450°F), slow furnace
cool, note appearance of twins in (b)

3(a)
100X



3(b)
100X

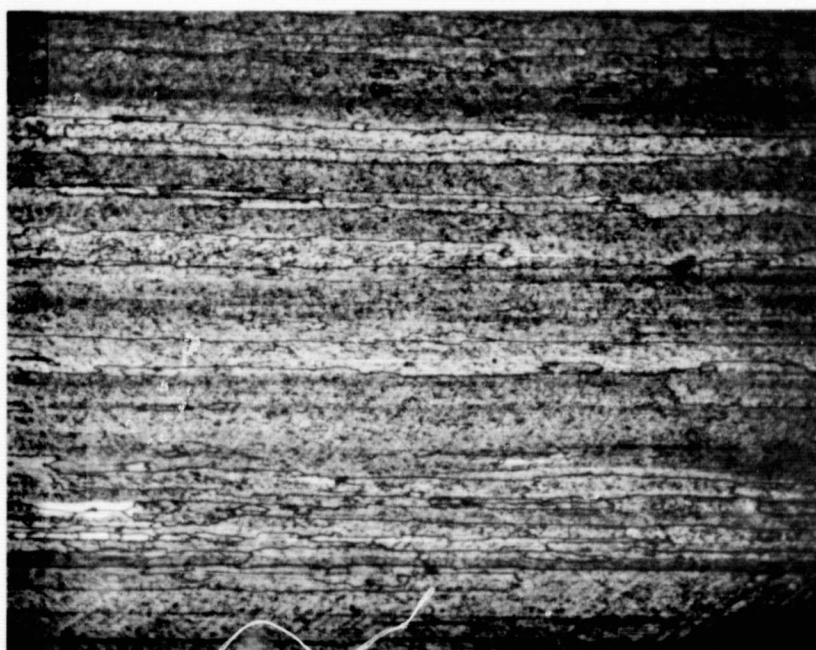


FIGURE 3: Cold roll plus anneal study, material 101, preannealed 1 hour at 2450°F, cold rolled longitudinally, longitudinal and short transverse plane shown

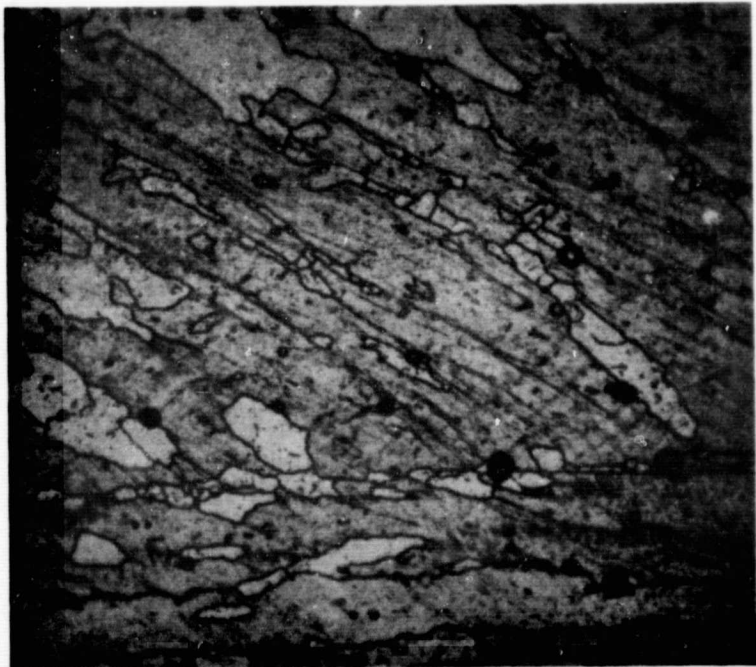
(a) 25% reduction, as rolled

(b) 25% reduction, then annealed at 538°C(1000°F), 1 hour, air cool

3(c)
100X



3(d)
500X



3(c,d) 25% reduction with 1 hour anneal at 1343°C(2450°F), air cool,
note recrystallized grains forming at angle to rolling direction in (d) .

4(a)
150X



4(b)
100X

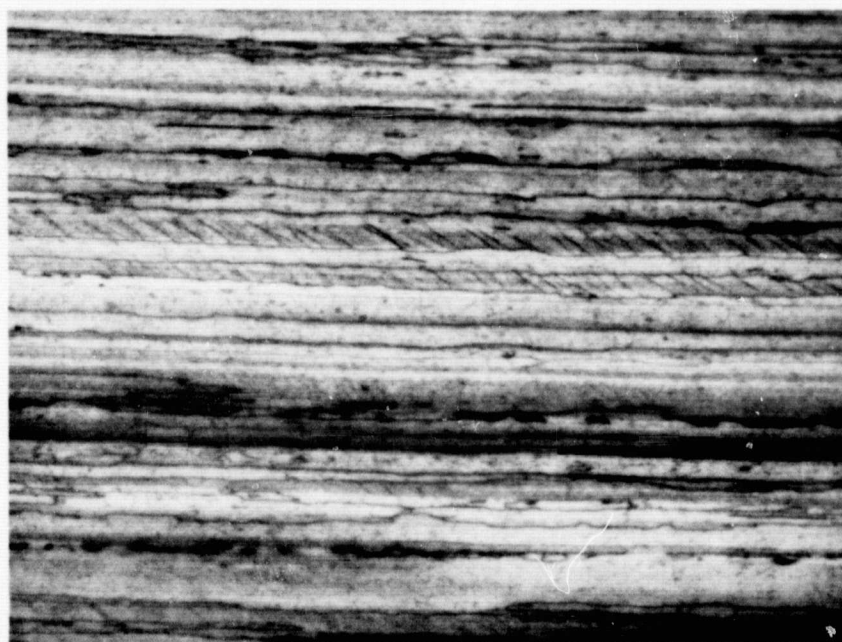
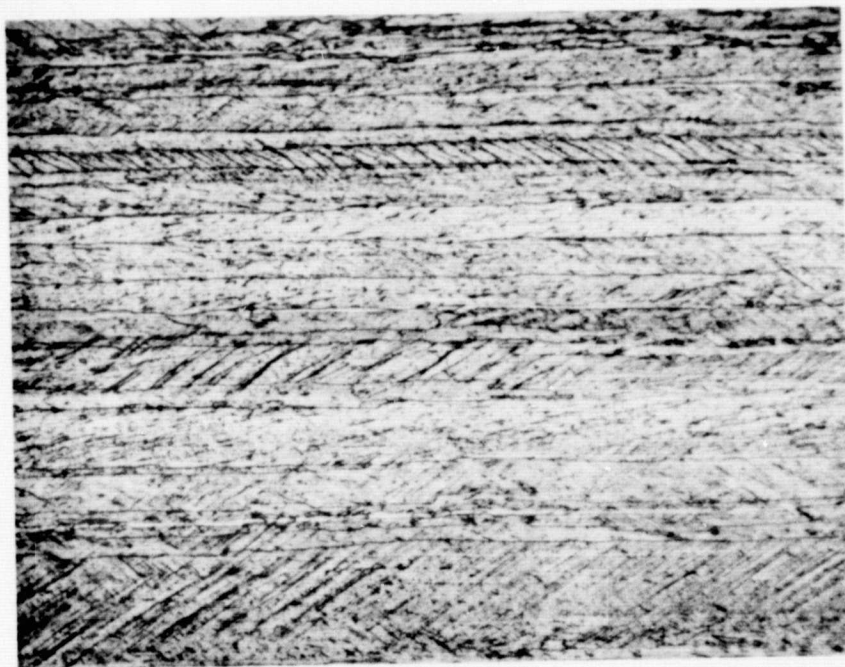
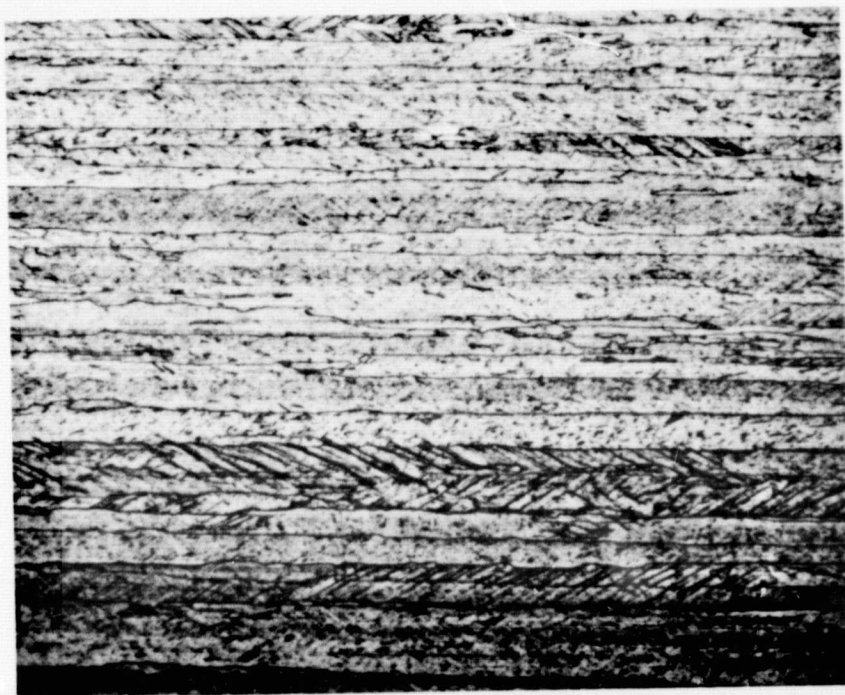


FIGURE 4: Cold roll plus anneal study, material 101, preannealed 1 hour at 2450°F, cold rolled longitudinally to 38% reduction, longitudinal and short transverse plane shown
(a) 38% reduction, as rolled,
(b) cold rolled with 1 hour anneal at 538°C(1000°F)

4(c)
150X

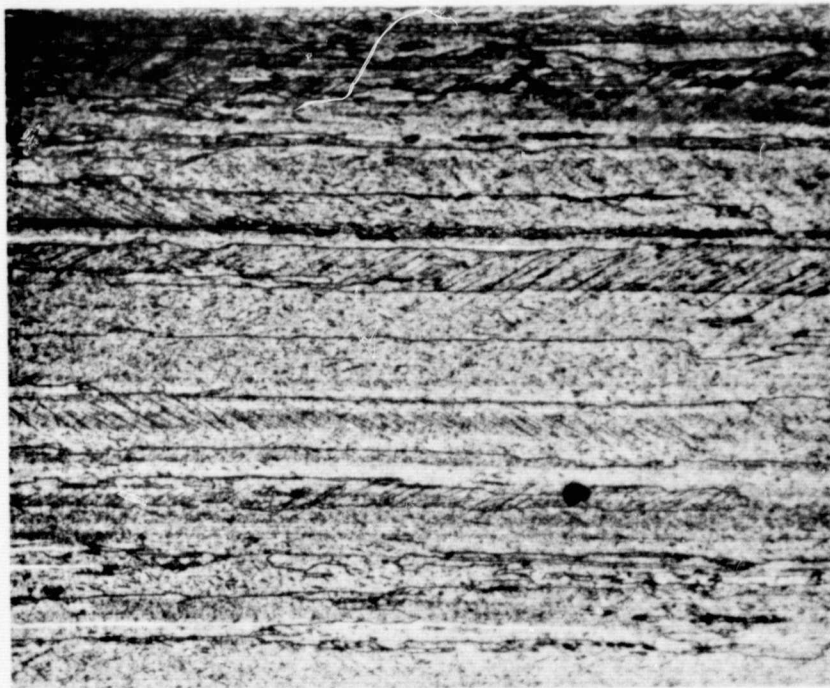


4(d)
150X



4(c) 38% reduction with 1 hour anneal at 760°C(1400°F)
4(d) 38% reduction with 1 hour anneal at 816°C(1500°F)

4(e)
150X

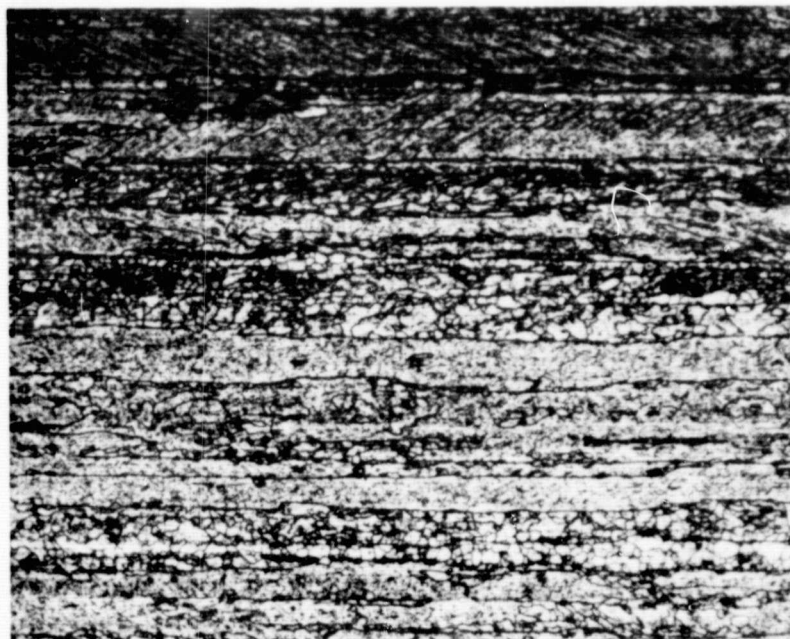


4(f)
150X

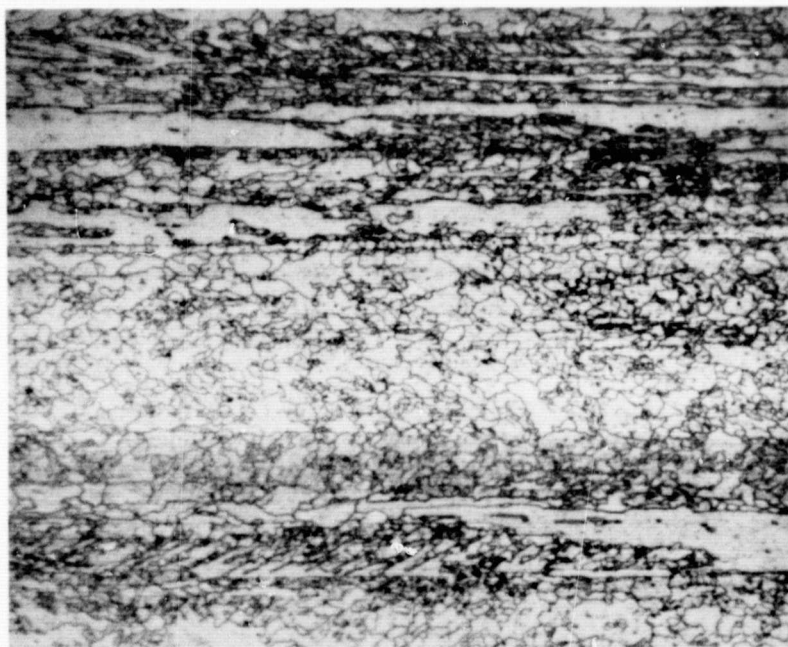


4(e) 38% reduction, then annealed 1 hour at 871°C(1600°F)
4(f) 38% reduction, annealed 1 hour at 927°C(1700°F), note appearance
of small grains

4(g)
150X



4(h)
150X

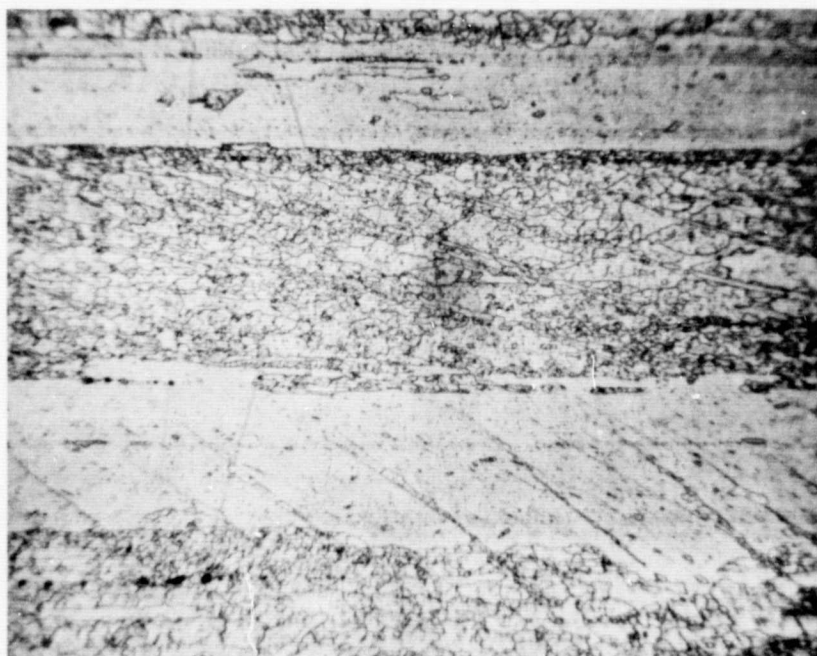


4(g) 38% reduction, annealed at 1038°C(1900°F), 1 hour
4(h) 38% reduction, annealed at 1093°C(2000°F), 1 hour

4(i)
200X

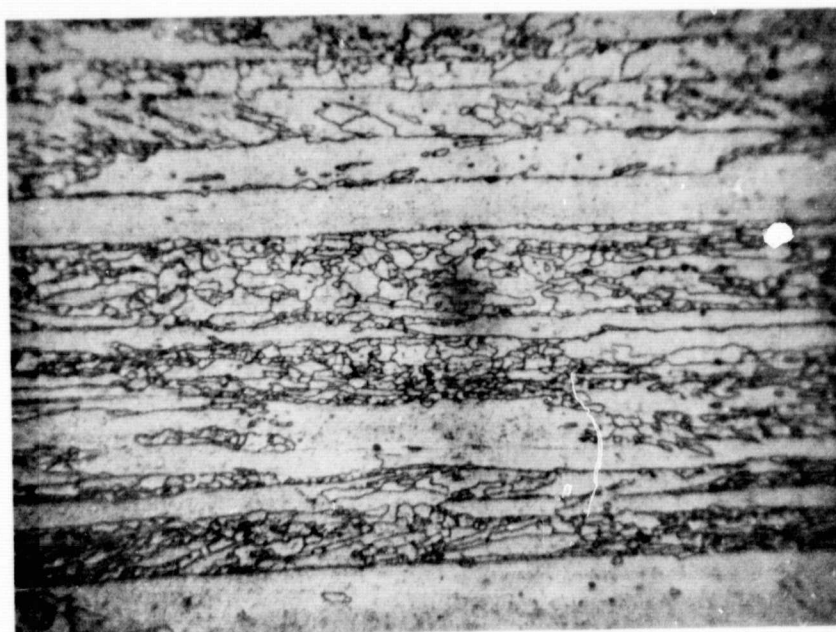


4(j)
200X



4(i) 38% reduction, annealed at 1204°C (2200°F), 1 hour
4(j) 38% reduction, annealed at 1260°C (2300°F), 1 hour, note
continued appearance of duplex grain structure with recrystallized
grains growing on slip line traces

4(k)
200X

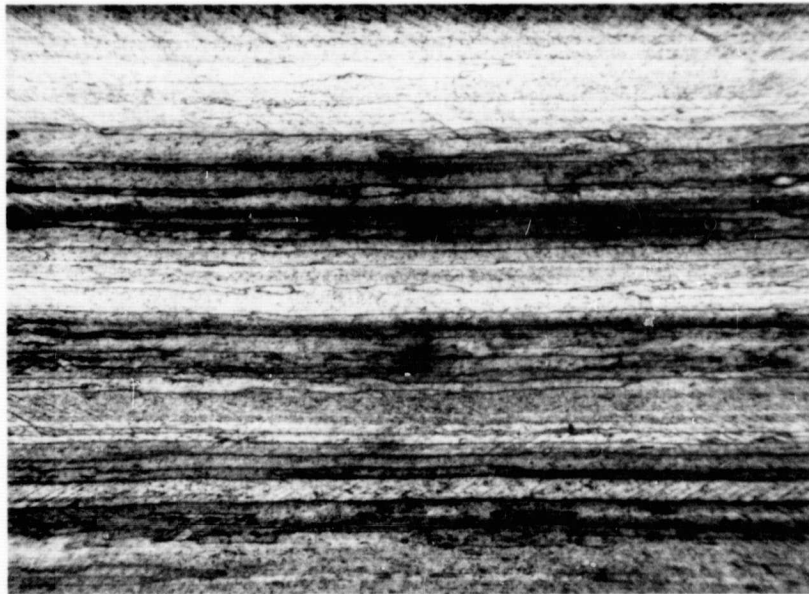


4(l)
200X



4(k) 38% reduction, annealed 1 hour at 1316°C(2400°F)
4(l) 38% reduction, annealed 1 hour at 1343°C(2450°F)

5(a)
100X



5(b)
500X

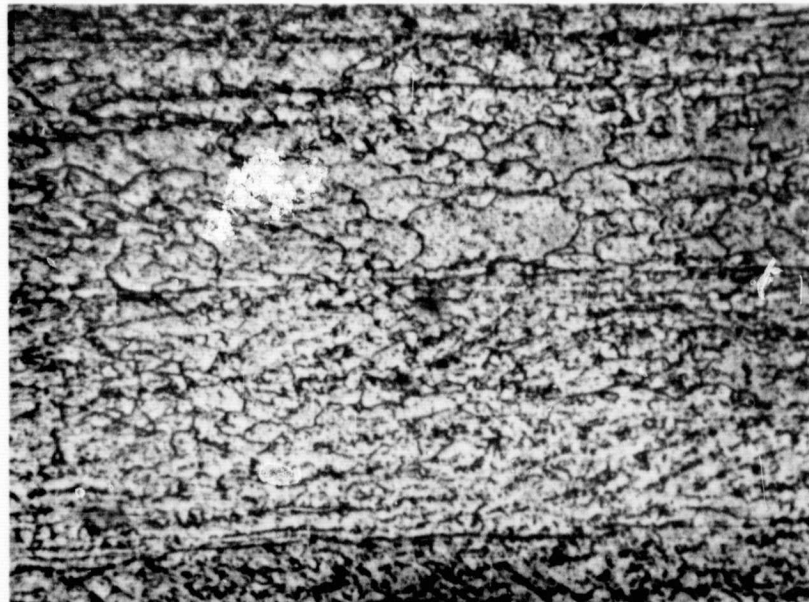
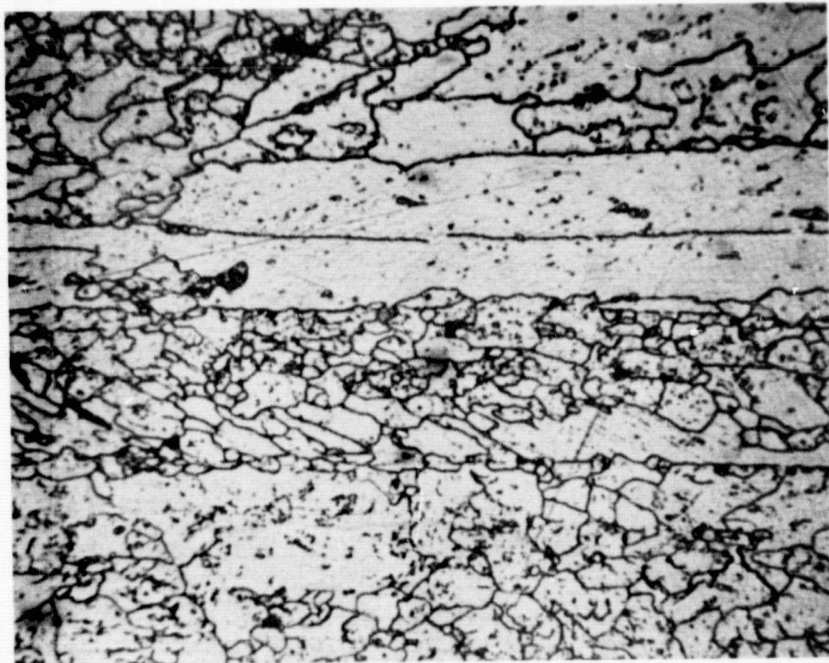
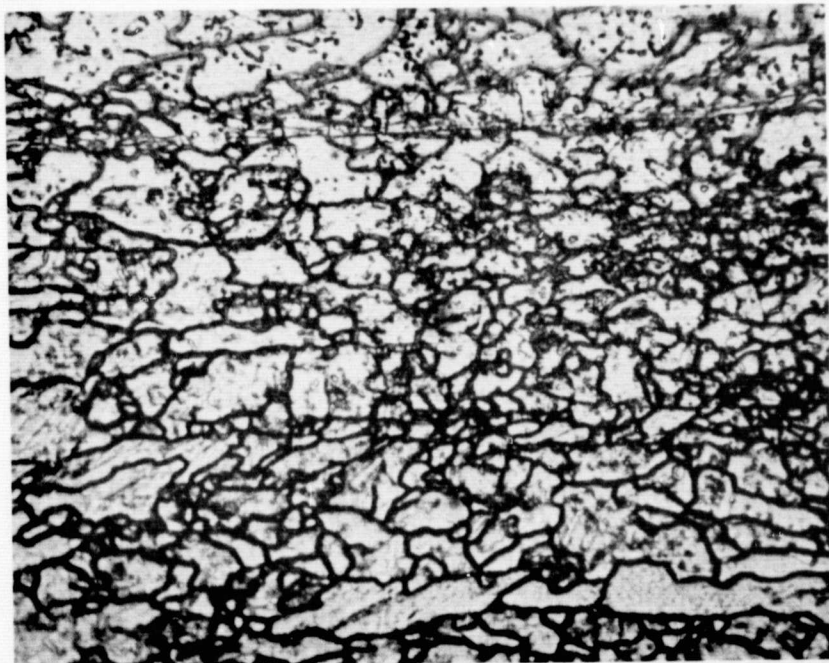


FIGURE 5: Cold work plus anneal study, material 101, preannealed 1 hour at 2450°F, cold rolled longitudinally to 50% reduction, longitudinal and short transverse plane shown
(a) as rolled, no anneal
(b) 50% reduction with 1 hour anneal at 871°C(1600°F)

5(c)
750X

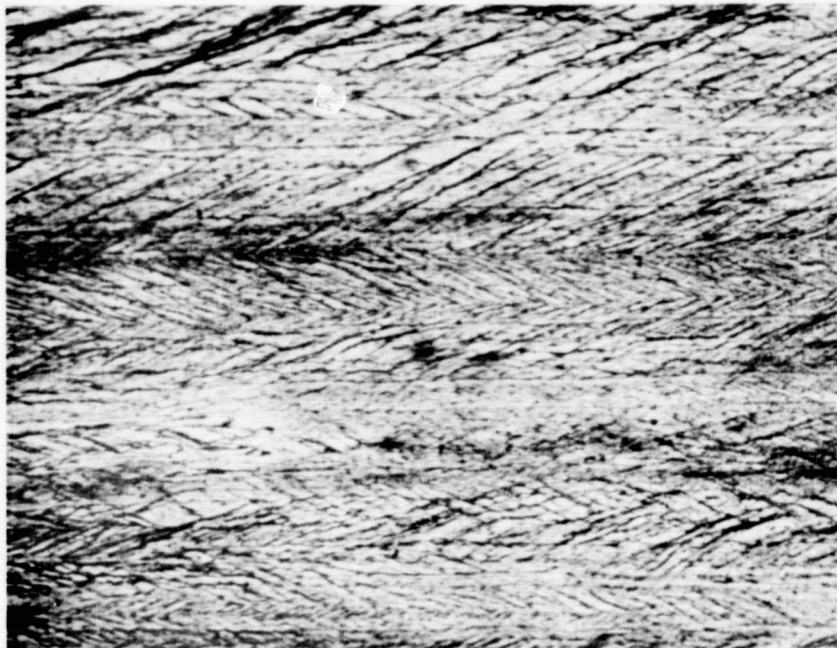


5(d)
750X



5(c) 50% reduction, annealed 1 hour at 1093°C(2000°F)
5(d) 50% reduction, annealed 1 hour at 1343°C(2450°F)

6(a)
250X

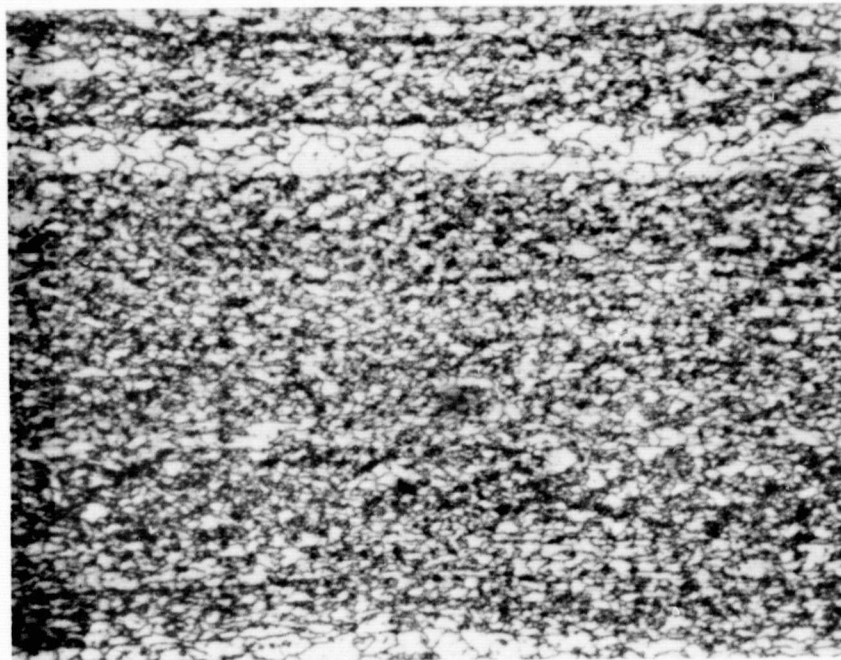


6(b)
250X

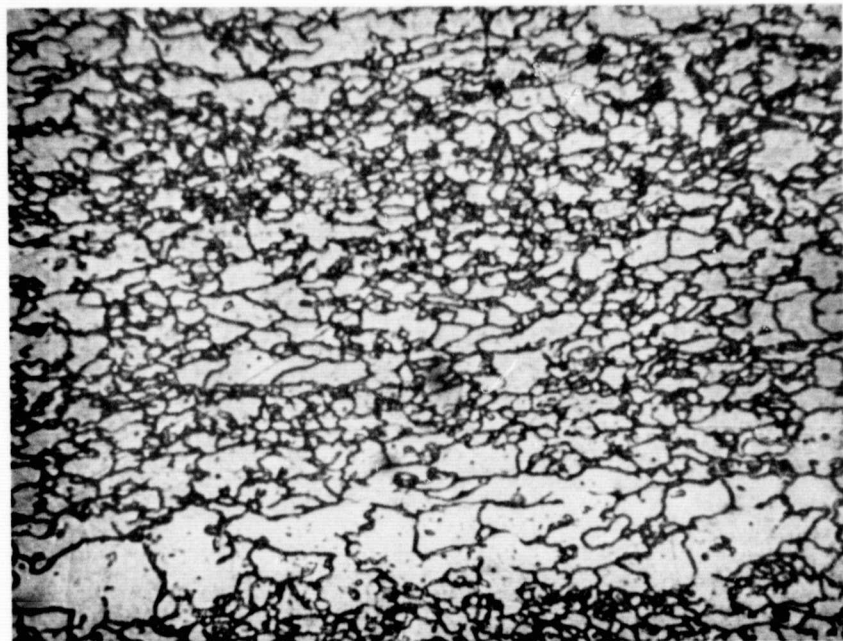


FIGURE 6: Cold work plus anneal study, material 101, preannealed 1 hour at 2450°F, cold rolled longitudinally to 73% reduction, longitudinal and short transverse plane shown
(a) as rolled, no anneal
(b) 73% reduction, with anneal at 538°C(1000°F), 1 hour

6(c)
250X

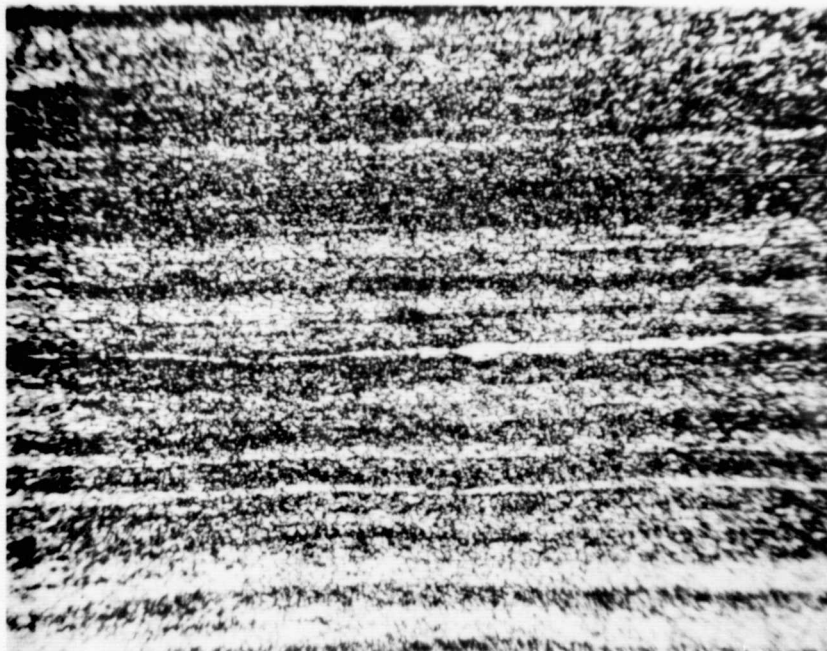


6(d)
750X

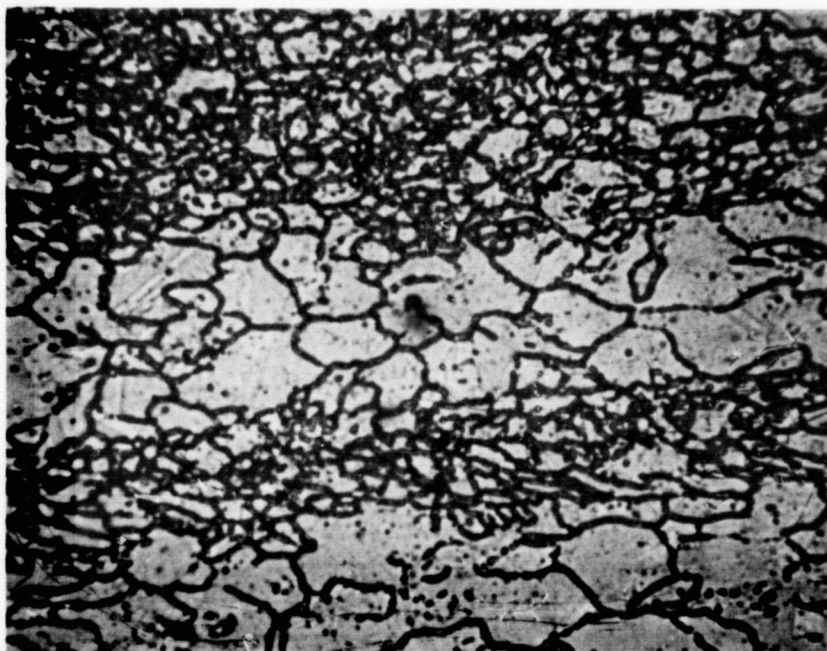


6(c,d) after 73% reduction, annealed at $1093^{\circ}\text{C}(2000^{\circ}\text{F})$ 1 hour
showing nearly equiaxed grain structure developing

6(e)
100X

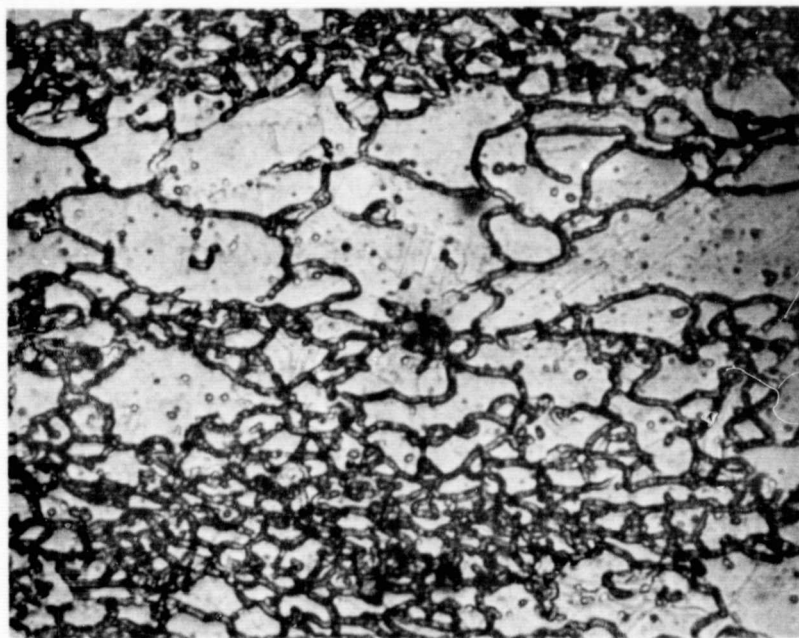


6(f)
750X

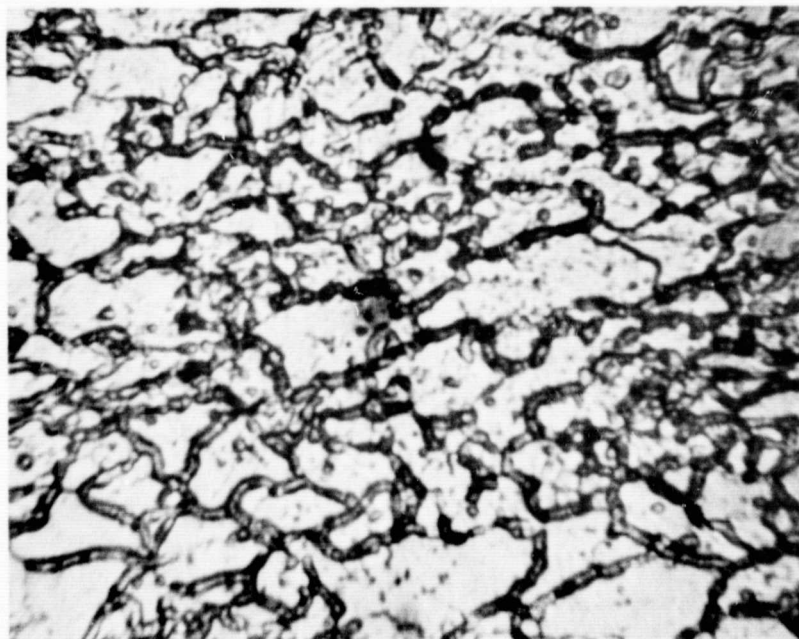


6(e,f) 73% reduction, annealed at 1343°C(2450°F), 1 hour, different magnifications showing equiaxed structure

6(g)
1000X

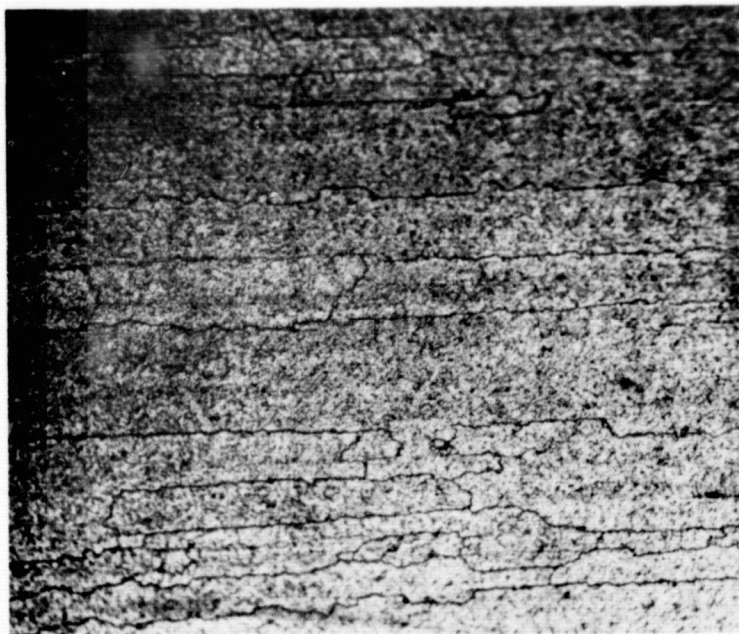


6(h)
2000X



6(g,h) 73% reduction, annealed at 1343°C(2450°F), 1 hour, note equiaxed
veinous network of grains at the higher magnifications

7(a)
100X



7(b)
75X



FIGURE 7: Cold work plus anneal study, material 204B, preannealed 1 hour at 2450°F, cold rolled in transverse direction, longitudinal and short transverse plane shown
(a) cross rolled to 12% reduction, annealed 1 hour at 1343°C(2450°F)
(b) cross rolled to 15% reduction, annealed 1 hour at 1343°C(2450°F)

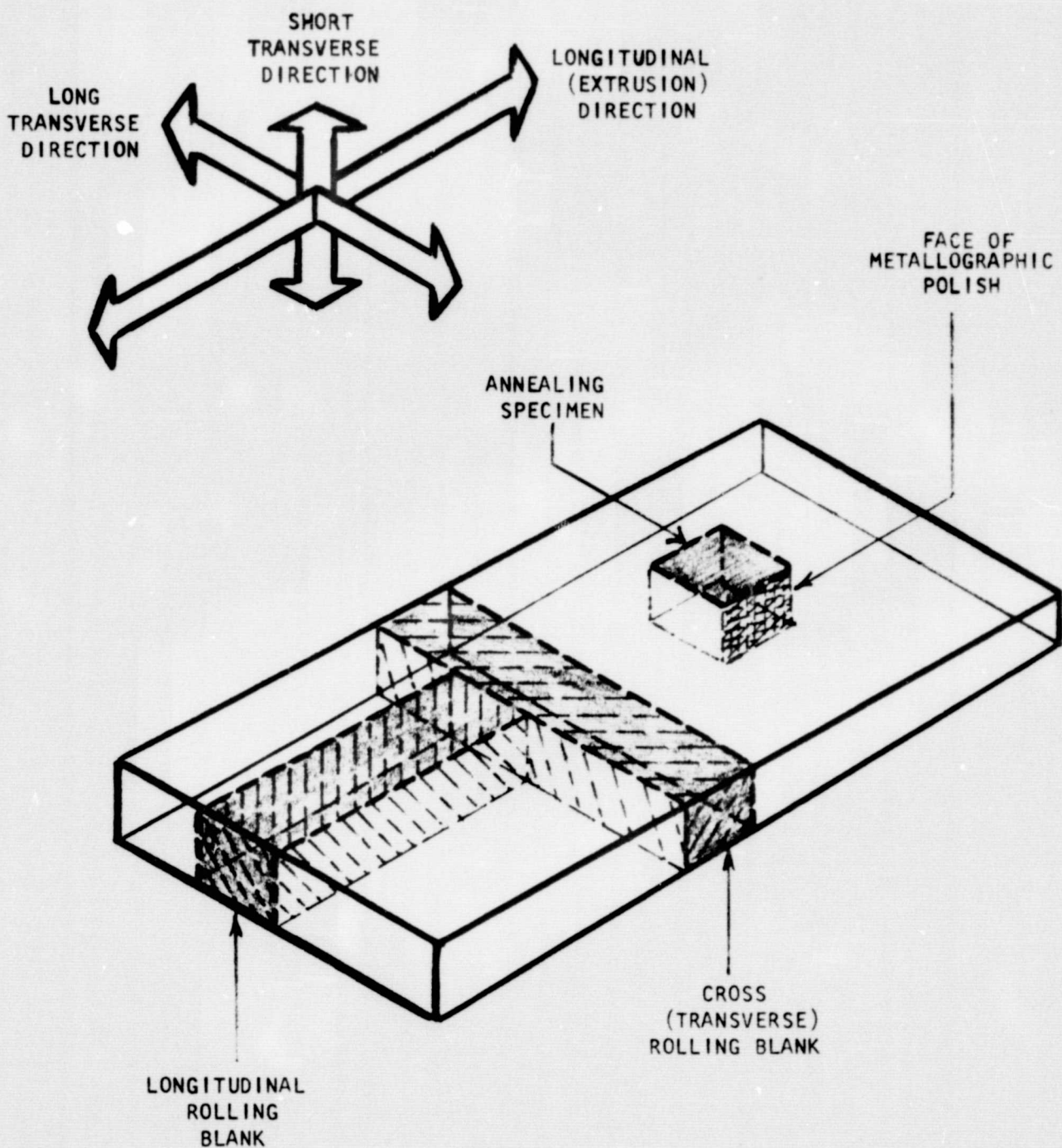


FIGURE 8 : SCHEMATIC ILLUSTRATION DEFINING VARIOUS DIRECTIONS WITH RELATION TO THE EXTRUSION DIRECTION, AND THE LOCATION OF THE VARIOUS BLANKS AND SPECIMENS CUT FROM THE AS-RECEIVED EXTRUSION

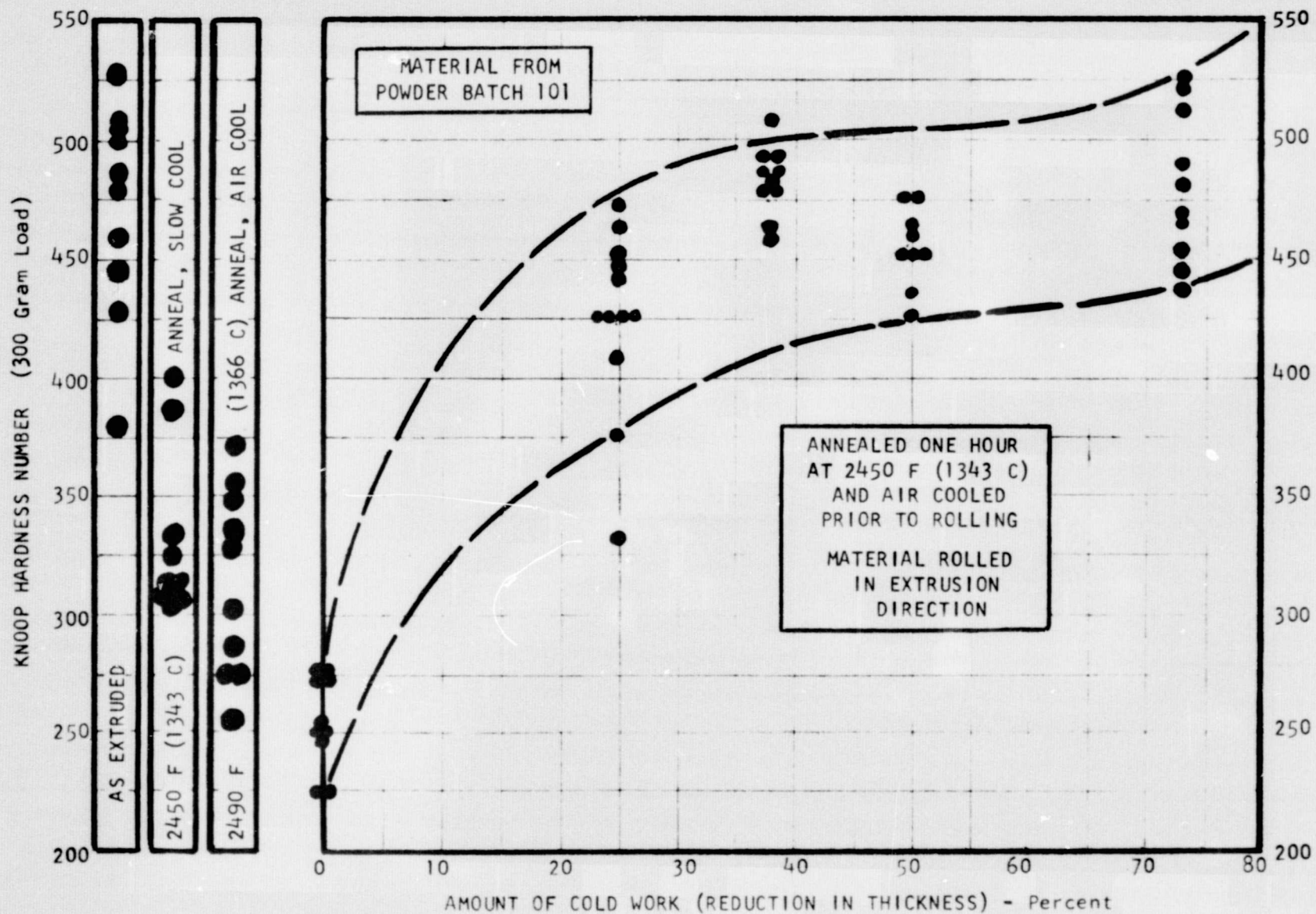


FIGURE 9 : AS-RECEIVED (AS-EXTRUDED), ANNEALED, AND COLD WORKED HARDNESS OF THE 101 MATERIAL

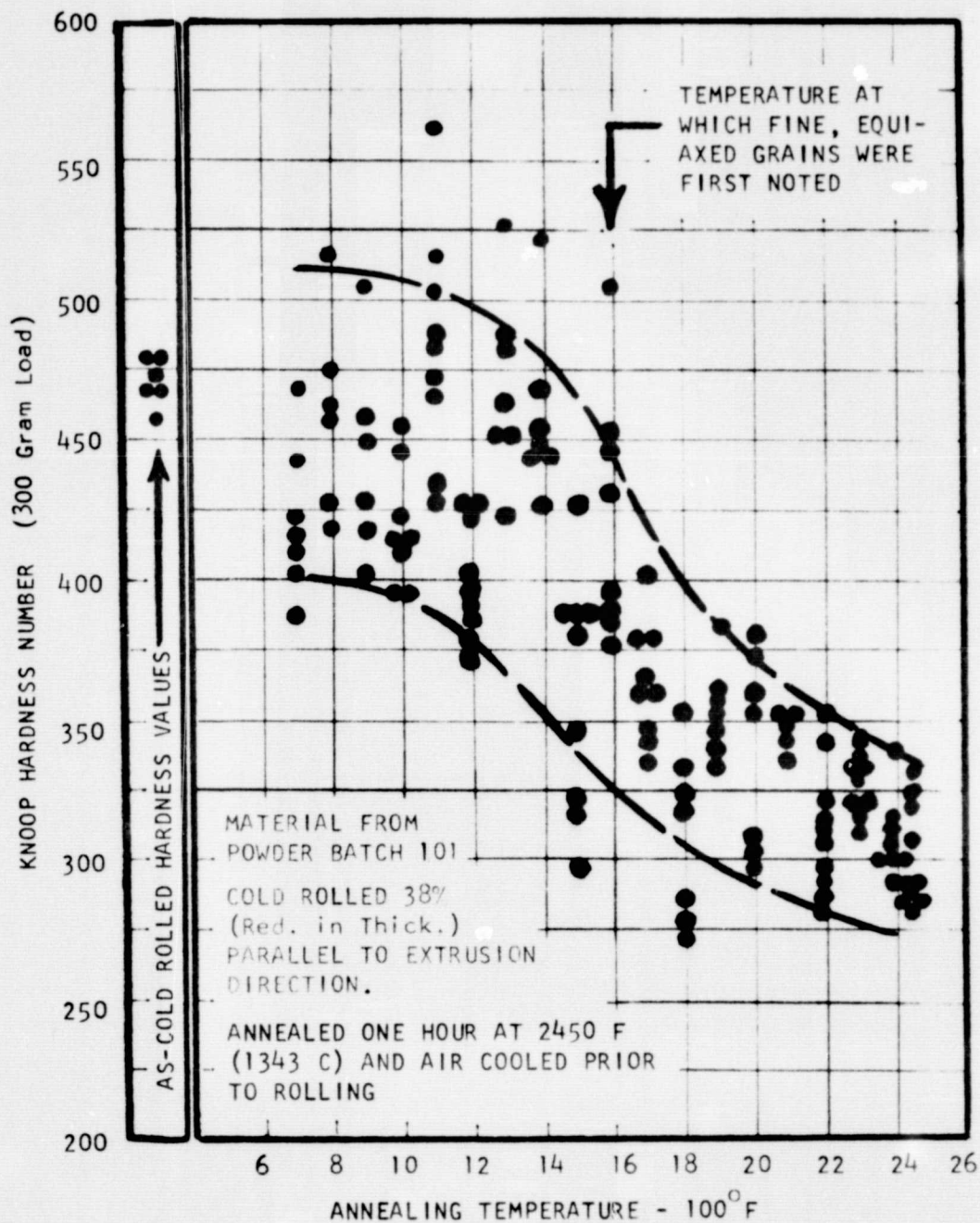


FIGURE 10: RECRYSTALLIZATION CURVE FOR THE 101 POWDER BATCH MATERIAL, COLD ROLLED 38% (Reduction in Thickness) PARALLEL TO THE EXTRUSION DIRECTION. (One hour annealing time at temperatures shown above; air cool from annealing temperature.)